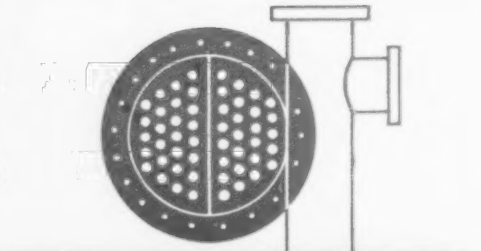
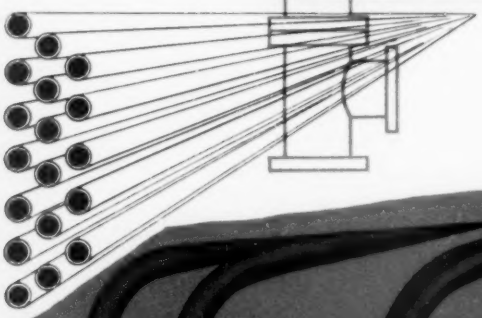
METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 60 No. 361 NOVEMBER, 1959 Monthly: Two Shillings and Sixpence



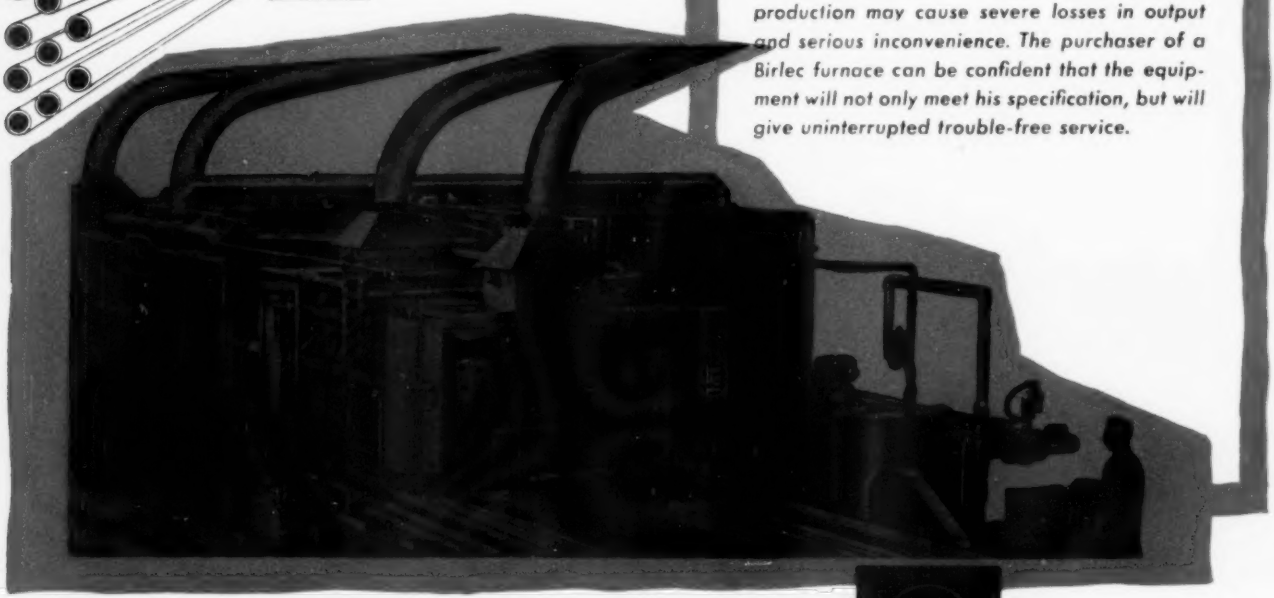
HIGH RATES OF EXCHANGE



Power Stations, Chemical Works and Food Processing Plant rely on heat exchangers whose efficiency and durability depend on high quality stainless steel tubes.

Such tubes are produced at the works of the Talbot Stead Tube Co. Ltd., who have recently installed a 6750 c.f.h. Birlec roller hearth furnace which anneals 40 tons of finished stainless steel tubing each week. The furnace works continuously for 5 days out of 7, and allowing for interpass work, the weekly throughput averages 100 tons, in sizes ranging from 1"—8" dia. and thicknesses from 18 gauge to $\frac{3}{8}$". The furnace has six clutches and two brakes which permits it to be controlled remotely by electronics for any required operating sequence.

**The cost of a furnace is not necessarily its purchase price. Even a brief interruption in production may cause severe losses in output and serious inconvenience. The purchaser of a Birlec furnace can be confident that the equipment will not only meet his specification, but will give uninterrupted trouble-free service.*



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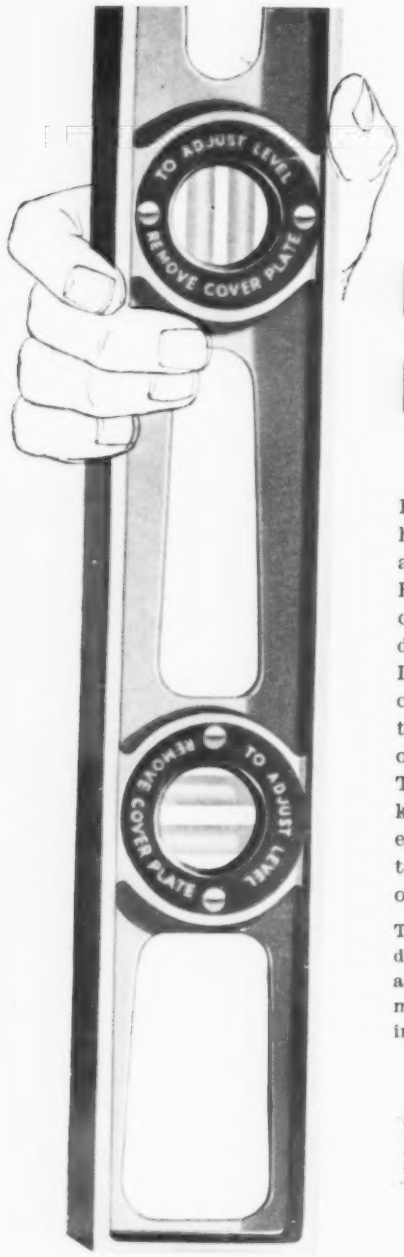


In hand but out of mind

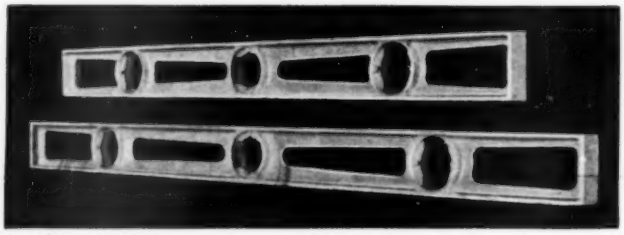
Picture a builder, his hand on the level, his eye on the bubble. How rarely does he take an interest in the spirit level's casing! How rarely does he need to when, like so many other familiar things around us, its stability depends on a Birmal casting.

Dependable Birmal! For more than 50 years Birmal craftsmen have been producing castings to support, to enclose, to provide the very foundation of a host of well-known products. Their technical skill and great experience keep Birmal ahead in foundry practice . . . ensure that Birmal castings will continue to be relied on wherever the quality of castings counts.

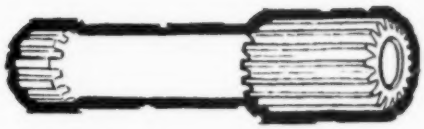
The Birmal foundries produce sand and gravity die castings in aluminium and magnesium alloys, and pressure die castings in aluminium, magnesium and zinc alloys for use in practically every industry.

BIRMAL

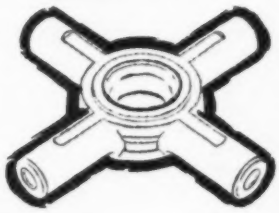
Pressure die cast in aluminium alloy for John Rabone & Sons Ltd.

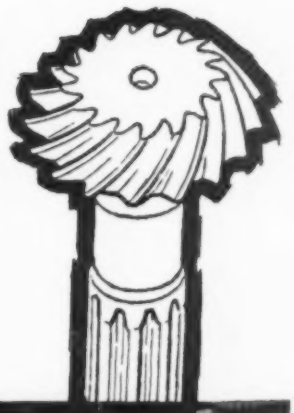


Birmingham Aluminium Casting (1903) Co. Ltd Birmid Works Smethwick 40 Staffs

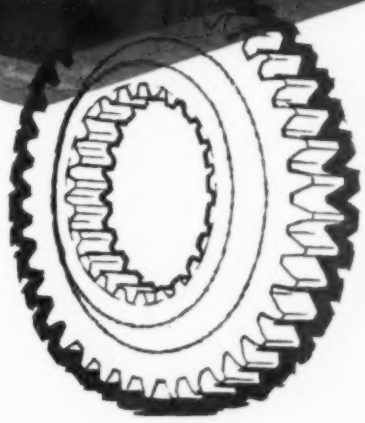


WILD BARFIELD





GAS CARBURISING



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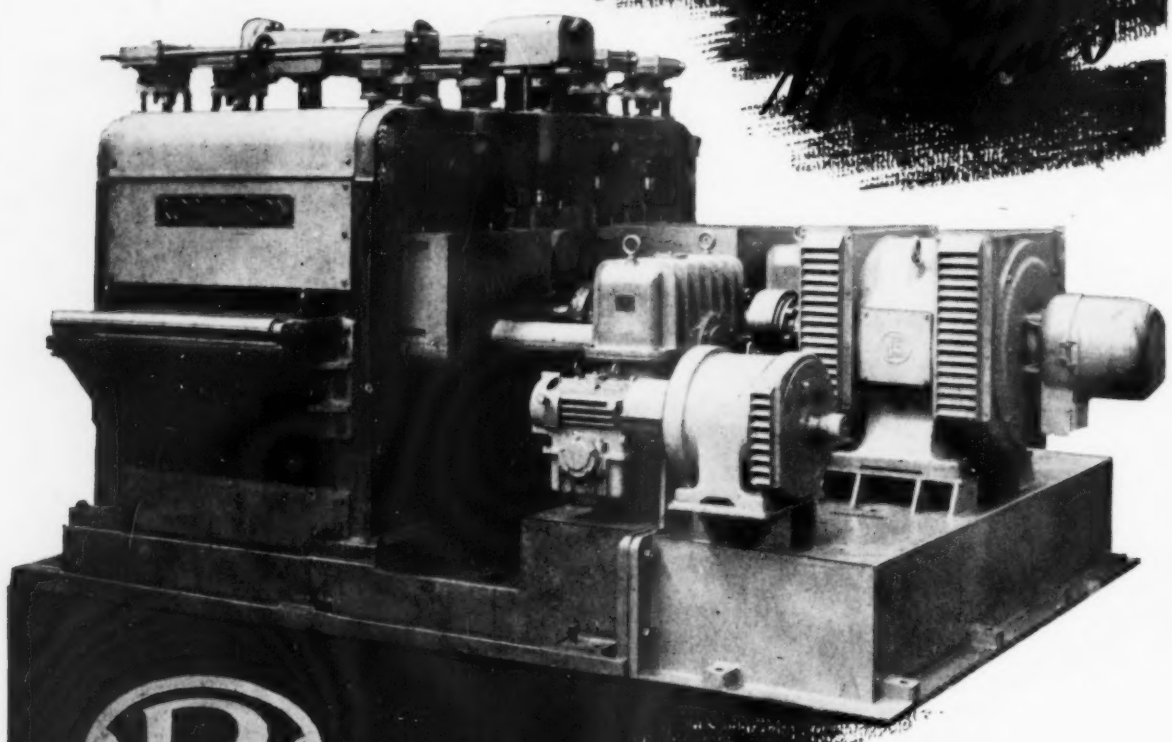

Depicted above is a No. 1653 Torrington Slab Milling Machine for overhauling non-ferrous strip up to 30" wide and $\frac{3}{4}$" thick, milling one face at a time.

The cutting speeds are variable up to 45ft/minute dependent on the material and general requirements.

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Complete with Motor Driven Positive Air Blower, two Gas Blast Blow Pipes and one Gas Blast Burner. Suitable for Tube bending up to 1 in. diameter.

Size of Hearth, 20 in. × 20 in. × 4 in.

Gas Consumption, 200 cu. ft. per hour maximum.

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Suitable for temperatures up to 900° C.

Obtainable in the following sizes :
8 in. dia. × 8 in. deep, gas consumption 300 cu. ft./hr.
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10 in. dia. × 12 in. deep, gas consumption 400 cu. ft./hr.

Time to heat up—1½ hours.

Manufactured by

KASENIT LTD

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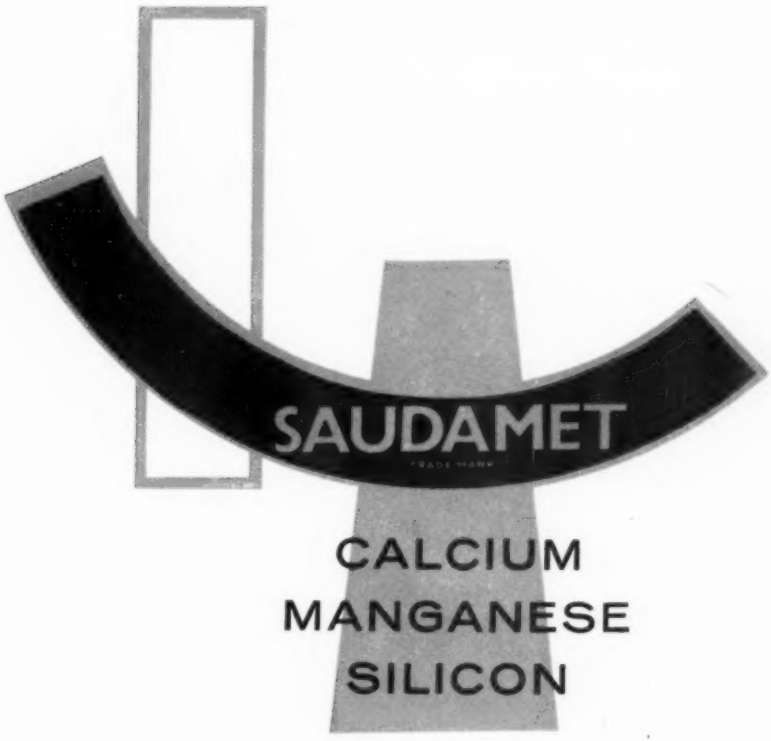

for clean steel . . . ductile steel . . . and better castability

Calcium Manganese Silicon is used as a ladle addition in the open hearth process and as both a furnace and a ladle addition in electric steel making. This combination of elements, which includes two extremely active and strong deoxidizers, produces exceptionally clean steel. It is especially helpful in the production of steel castings: in this application the combination of calcium, silicon, and manganese is effective in breaking up the harmful chain-type inclusions commonly associated with poor ductility. Castings treated with Calcium Manganese Silicon show exceptional uniformity, high ductility and freedom from internal tears.

In basic electric furnace practice, Calcium Manganese Silicon is widely used for quick deoxidation of the metal. In all electric steel, it aids in overcoming low ductility often caused by high aluminium additions. Stainless steels and certain other high-alloy steels have their castability improved by treatments with this alloy.

More information about this excellent alloy (Ca 16-20%, *Mn* 14-18%, *Si* 55-60%) *can be obtained from Union Carbide Limited, Alloys Division,* 103 *Mount Street, London, W.*1. (*Telephone HYDe Park* 5361).

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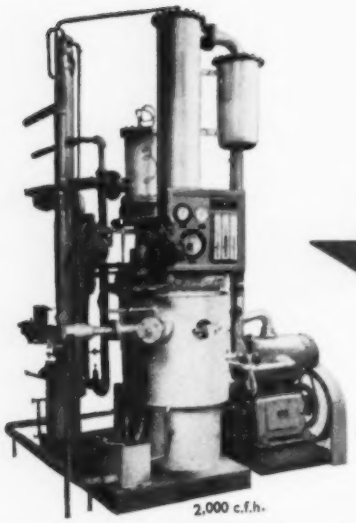
Atmosphere generators with a wide range of applications in many industries. In the metal industry for bright annealing, brazing, sintering, gas carburising, bright hardening, etc.



2,000 c.f.h.

GmvL Ammonia Cracker

For bright treatment of stainless acid resisting and heat resisting steels, and for brazing and sintering generally.

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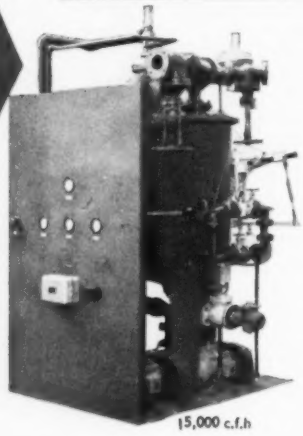
A highly efficient and compact producer of inert gas, operating on standard gaseous fuels and distillate grades of fuel oils.



15,000 c.f.h

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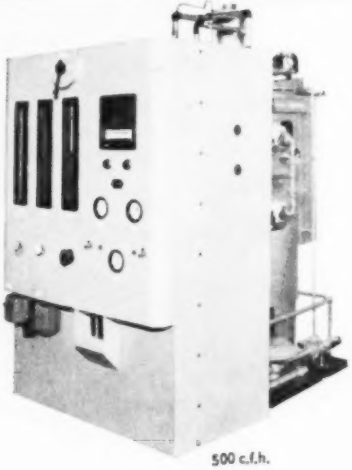
For gas carburising, dry cyanide treatment, etc. Automatic response to variations in demand while maintaining correct gas/air ratio.



500 c.f.h.

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A SUBSIDIARY COMPANY OF GIBBONS BROS. LTD.

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'Phone : Edgbaston 4269

Characteristic of British Craftsmanship is this handsomely worked pistol. Made in the 16th Century from steel and wood it is a fine example of the work of that period and can be seen in the Victoria and Albert Museum.

Britain is proud of her craftsmen; throughout the years their skilled workmanship has earned a reputation for Britain that is second to none. The historical associations of steel in England chronicled through the ages, show how dependent we have become upon its production. Today its manifold uses in industry and everyday life have become legion—civilisation itself is woven over a framework of iron and steel. Hallamshire high grade Alloy and Carbon steels are an integral part of Britain's traditional good name.

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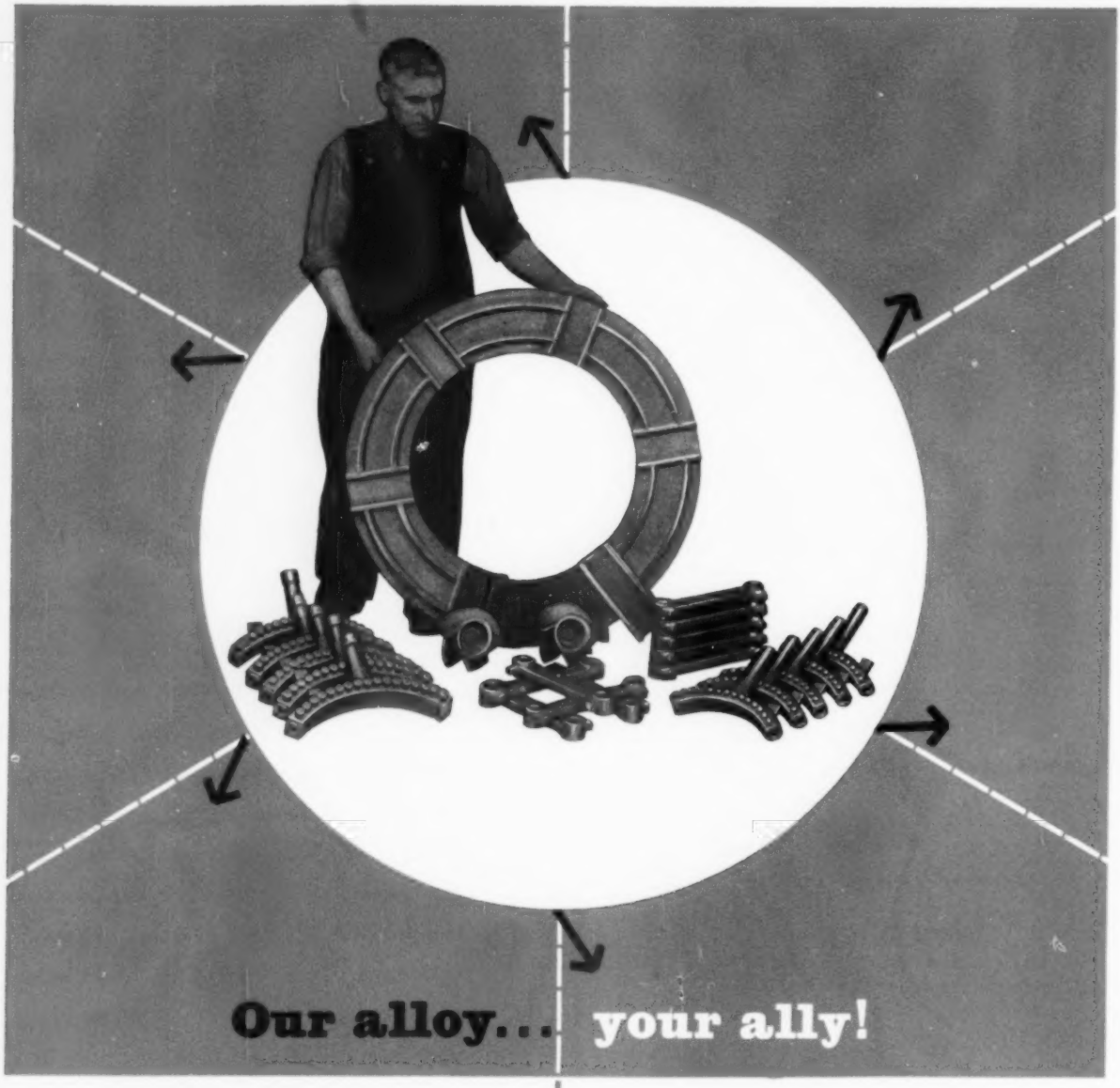
BARS · SHEETS · PLATES · WIRE RODS

THE HALLAMSHIRE STEEL COMPANY LIMITED · SHEFFIELD 3 · ENGLAND
Telephone: Sheffield 24304 (5 lines) Telegrams: Hallamsteel Sheffield



The Thompson L'Hospied service embodies much more than the production of accurate and consistently fine heat-resisting castings. Through their close association with the furnace industry they have first-hand knowledge of its requirements and unequalled facilities for testing under actual conditions.

Added to these unique advantages is the specialised knowledge acquired during over twenty-five years in the trade. Thompson L'Hospied advice on alloys and design is widely sought and freely given to customers in every industry where heat or chemical treatment plays a part.

Thompson PIONEERS OF T'H HEAT RESISTING CASTINGS **L'Hospied**

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THOMPSON L'HOSPIED & CO. LTD., STOURBRIDGE, ENGLAND.

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from experience!

From experience Gibbons "H.T 1" insulating refractory is the automatic choice of leading furnace manufacturers and users—its combination of low thermal conductivity with resistance to high temperatures has proved remarkably effective in minimising heat losses and raising furnace outputs. The Gibbons "H.T 1" is developed from more than 20 years' experience in the manufacture of insulating refractories and today an entirely new and original manufacturing technique enables us to offer material having unique physical properties at a price you can afford to pay.

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Refractory Insulating Concrete · Concrete Aggregates · Super-Plastic Jointing Cements · Surface Coating Cements

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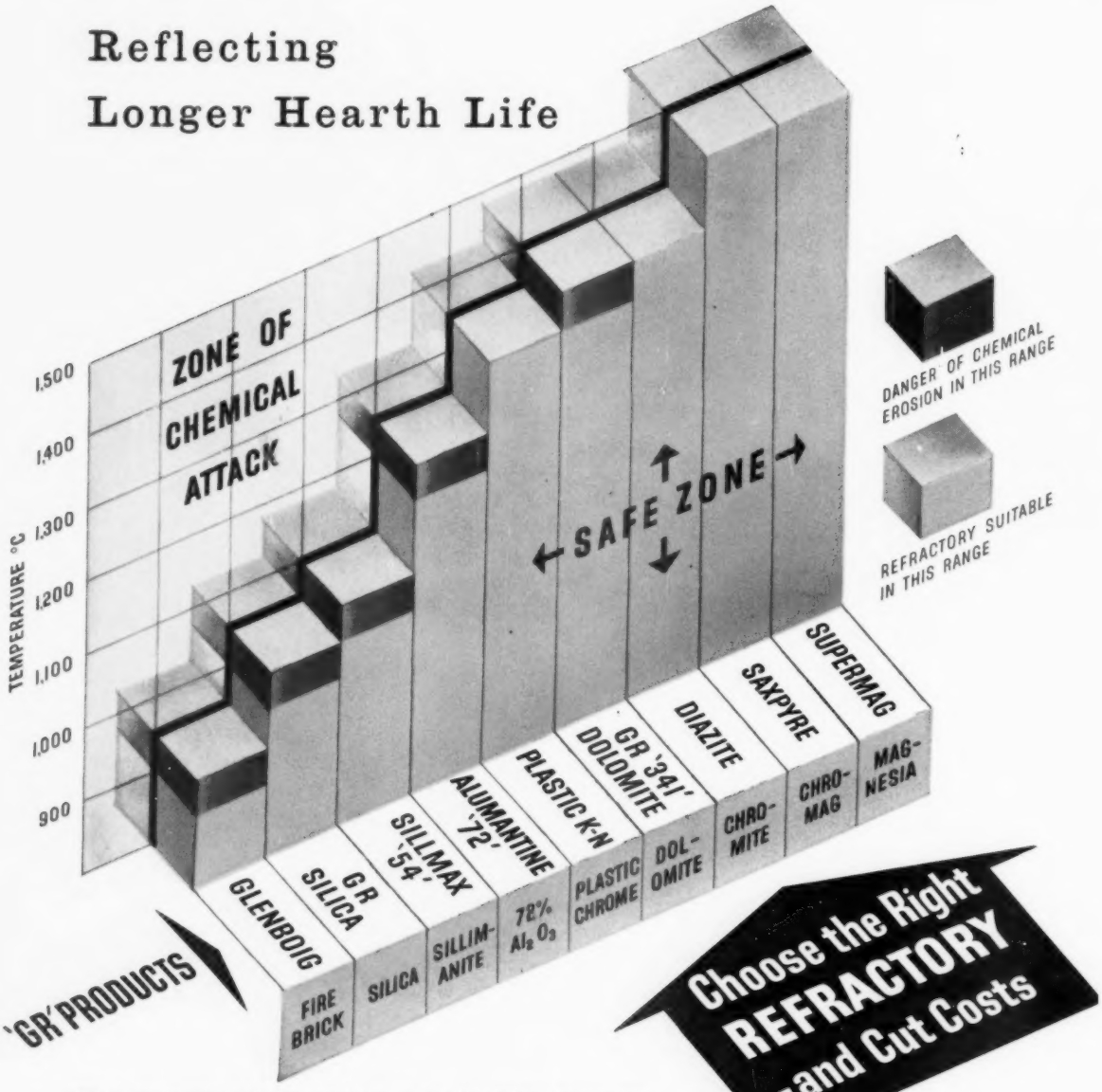
DO YOU OPERATE REHEATING FURNACES?

Reflecting Longer Hearth Life



The economical operation of reheating furnaces depends greatly upon the life of the refractory material selected for the hearth. Whether your immediate problem concerns in-and-out furnaces, continuous slab, end pusher, forging or bogi-type furnaces or one of the various types of soaking pits, it pays to let GR guide your selection of refractories. The GR Technical Service Department is available to advise on the correct choice of refractories for any specific application.

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Everything in Furnace Linings

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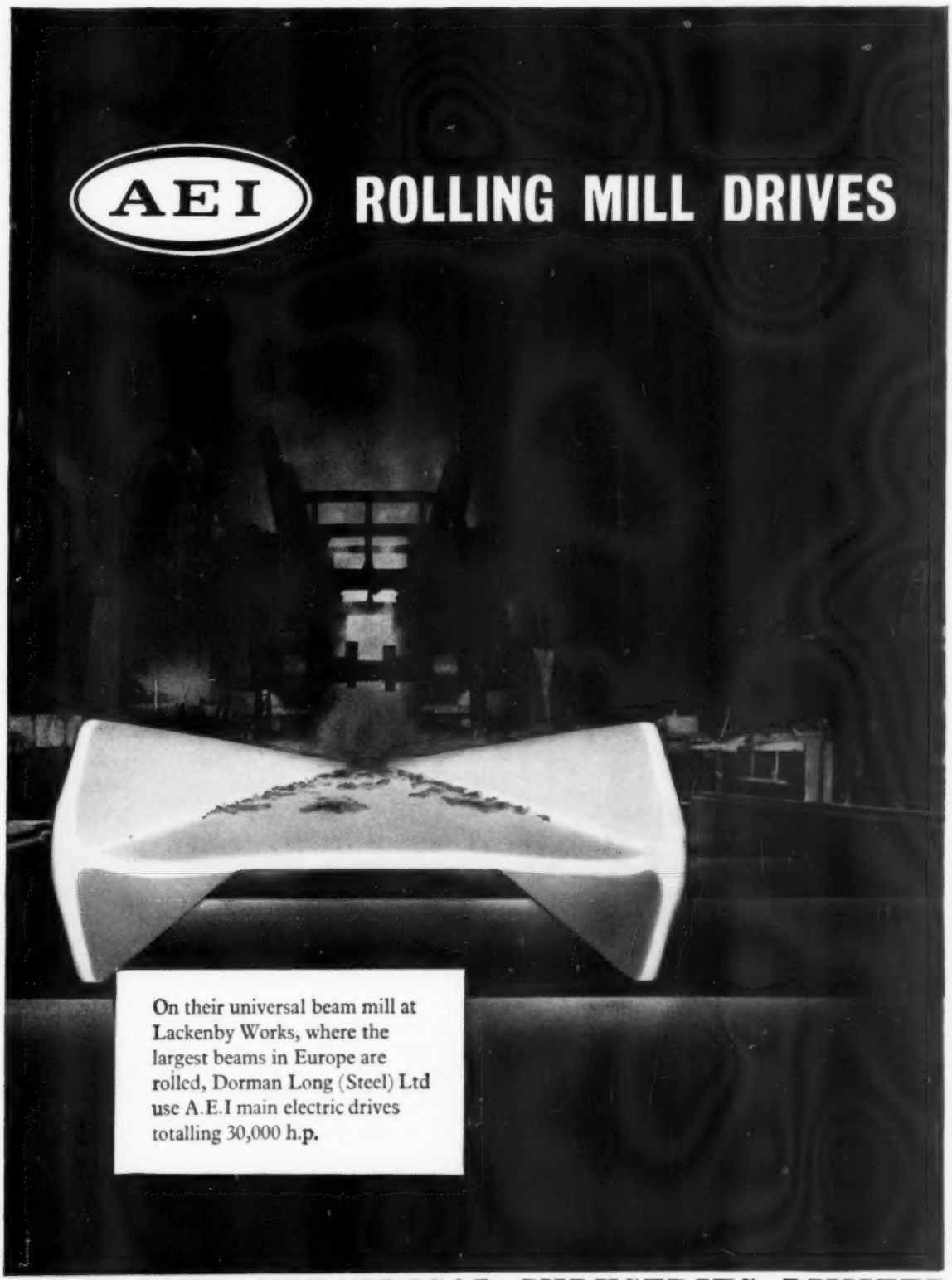

On their universal beam mill at Lackenby Works, where the largest beams in Europe are rolled, Dorman Long (Steel) Ltd use A.E.I main electric drives totalling 30,000 h.p.

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Rolls and Roll Lathes by Brightside for the 11" Continuous Bar Mill.

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THE BRIGHTSIDE FOUNDRY & ENGINEERING CO. LTD.

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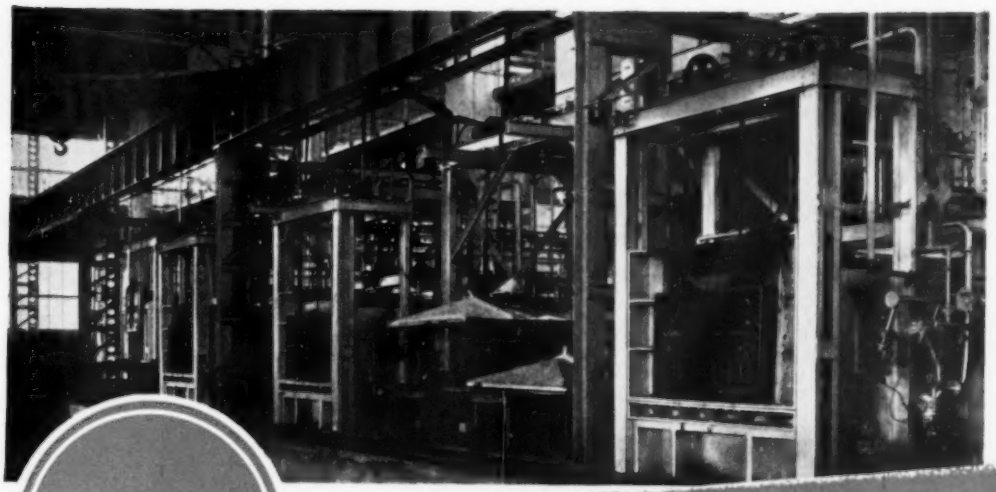
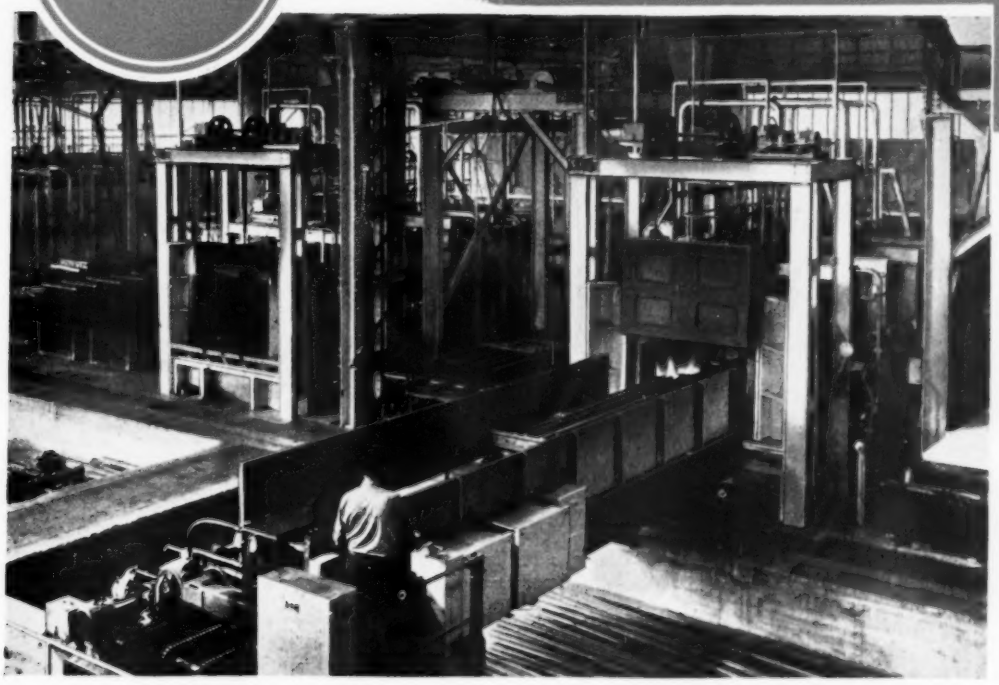


FURNACES AND QUENCHING EQUIPMENT.

PRIEST

BAR TREATMENT PLANT



FURNACES AND 3 PEEL CHARGING MACHINE.

We specialise in the design and construction of:—

Open Hearth Furnaces
Soaking Pits of all types
Continuous Multi-zone Bloom and Slab Re-heating Furnaces
Continuous Bogie type Ingot and Slab Heating Furnaces
Furnaces for Aluminium Melting, Coil Annealing and Slab Re-heating
Forge and Heat Treatment Furnaces
Stress Relieving Furnaces
Shipyard Plate and Bar Furnaces
Modern Lime Burning Kilns

The illustrations show a battery of 3 Heat Treatment Furnaces, one with recirculation, 5 ton 3 peel charging machine, fixed and mobile quenching equipment. This plant, installed at the Stocksbridge Works of Samuel Fox & Company Limited, also includes two Bogie type Furnaces with special transfer charging machine.

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The last word in Furnace design

PRIEST FURNACES LIMITED · LONGLANDS · MIDDLESBROUGH

also at TELEGRAPH BUILDINGS HIGH STREET SHEFFIELD

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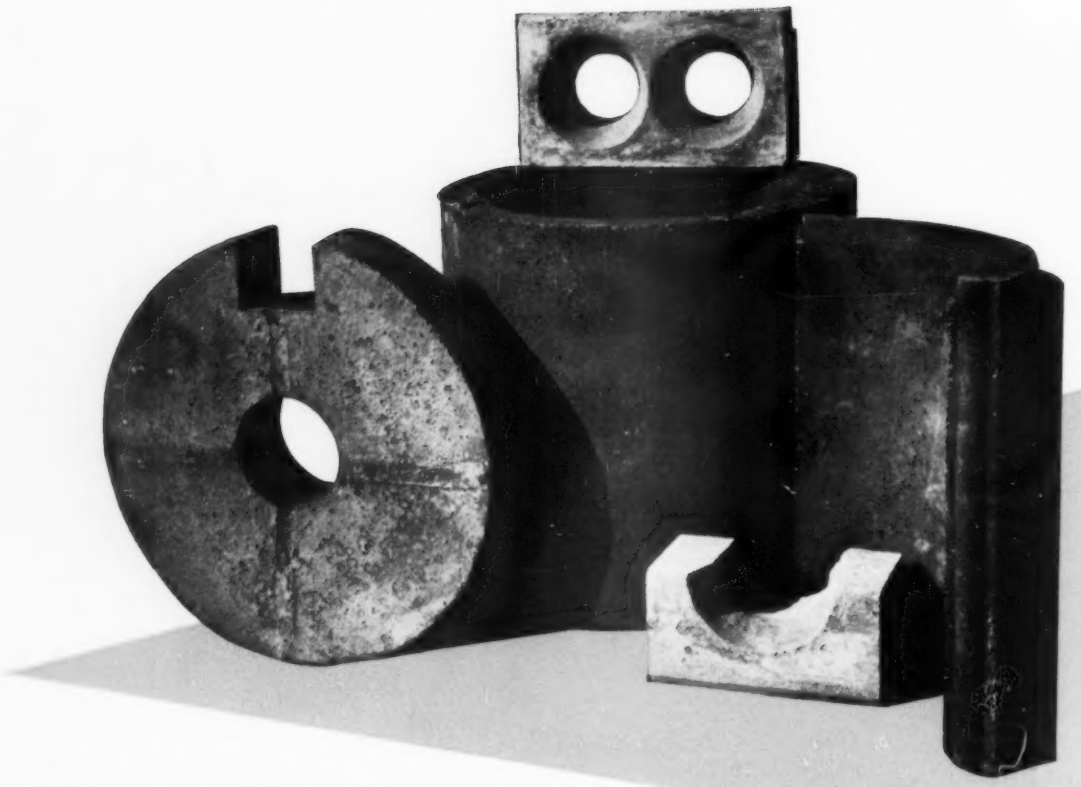
STEIN *Refractories*



REFRACTORY CONCRETE

Refractory Castables are poured and cast like structural Concrete, and being readily available are finding many applications, particularly for preparation of special shapes at site.

	HOW SUPPLIED	REFRACTORINESS	TYPE OF SETTING	THERMAL HARDENING TEMPERATURE	MAXIMUM TEMPERATURE OF USE	lbs./Cu. Ft.
Stein Refractory Concrete	Dry	1420°C	Hydraulic	1200°C	1350°C	120
Stein Super Refractory Concrete No. 16	Dry	+1750°C	Hydraulic	1200°C	1600°C	160
No. 17	Dry	+1750°C	Hydraulic	1200°C	1700°C	160
No. 18	Dry	+1750°C	Hydraulic	1200°C	1800°C	160
Stein Chrome Concrete	Dry	+1750°C	Hydraulic	1200°C	1500°C	180

Use our advisory service based on 70 years experience in the refractory field—it can improve your furnace efficiency. For further information write, phone or call:

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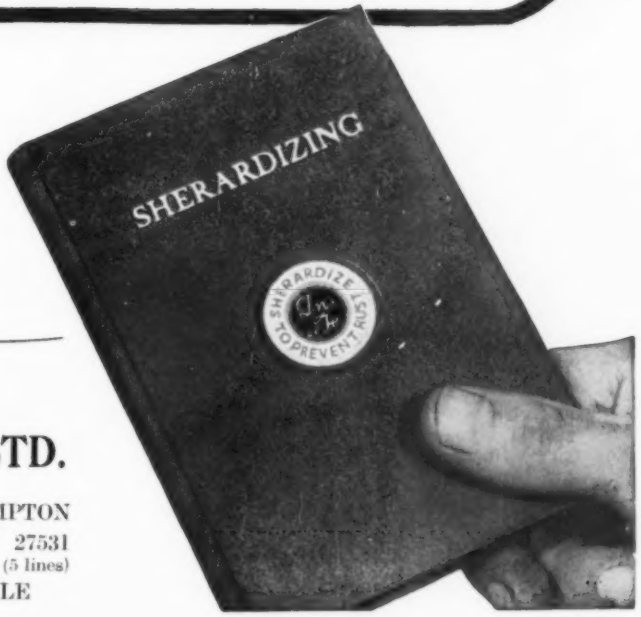

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TELEPHONE: WOLVERHAMPTON 27531
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ALSO AT LONDON AND ROCHDALE

Reining - HOT COOLING

Open Hearth Furnace Door Frame (Thermo syphon). Steam produced approximately 2,000 lbs./hr. per door frame.

This system of evaporative cooling, applicable to the water-cooled parts of all types of metallurgical furnaces has been fully developed and established in Germany during the last ten years by **Reining-Heisskühlung of Mulheim/Ruhr.**

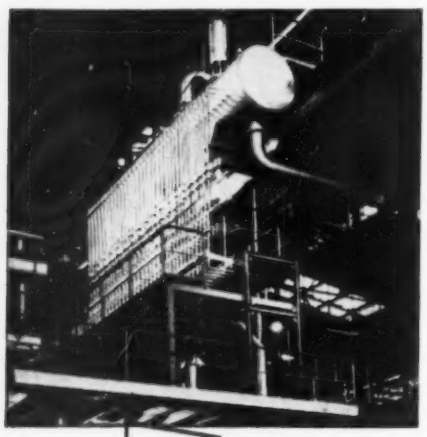
The Reining system is now being applied in Great Britain and throughout the Commonwealth exclusively

by:—

Stein & Atkinson

TYPICAL APPLICATIONS

Skid systems of 3 zone Continuous Reheating Furnaces (Forced circulation).
Steam produced approximately 20,000 lbs./hr. on 100 ton/hr. furnace.



1. Heat formerly lost in the cooling water is recovered in the form of saturated steam at any desired pressure. This heat recovery produces substantial fuel savings.

2. The quantity of cooling water required is limited to a make-up of boiler feed water corresponding to the quantity of steam consumed.

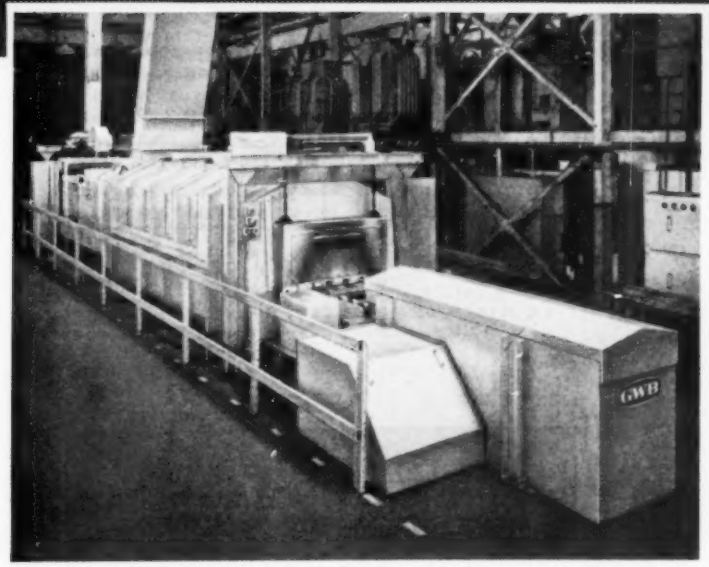
3. Failures of water-cooled parts due to blockage by deposits from untreated water are eliminated.

S & A

STEIN & ATKINSON LTD. LONDON

ALBION MOTORS AND G.W.B.

One of the oldest manufacturers of commercial motor vehicles in this country,
Albion Motors keep up to date in their manufacturing methods
by installing the latest and most efficient equipment available.
An example is the electrically heated Pusher Furnace supplied
by G.W.B. Furnaces Limited.



Overall view from charging end of 145 kw Pusher Type Normalising Furnace at Albion Motors Limited

APPLICATION

Various forgings such as main shafts, countershafts, pinion shafts and gear blanks are normalised by heating up to 900° C. and slow cooled down to 400/500° C.

AUTOMATIC OPERATION

The only labour required is for placing the components into the charge trays at the entrance end of the furnace. By pressing a button, the following cycle automatically takes place. (1) The entrance door rises. (2) The pusher ram propels the loaded tray into the furnace. (3) The ram returns. (4) Entrance door lowers. (5) Exit door rises. (6) Hydraulically operated go-getter pulls last tray from exit end of furnace on to a cross conveyor. The cross conveyor moves the normalised parts to the top of an inclined roller track where the tray is unloaded by a tipper, and then returns empty to the entrance end of the furnace.

OUTPUT

600 lbs. of normalised forgings per hour.

Over 25 years experience in electric furnace design at your disposal.

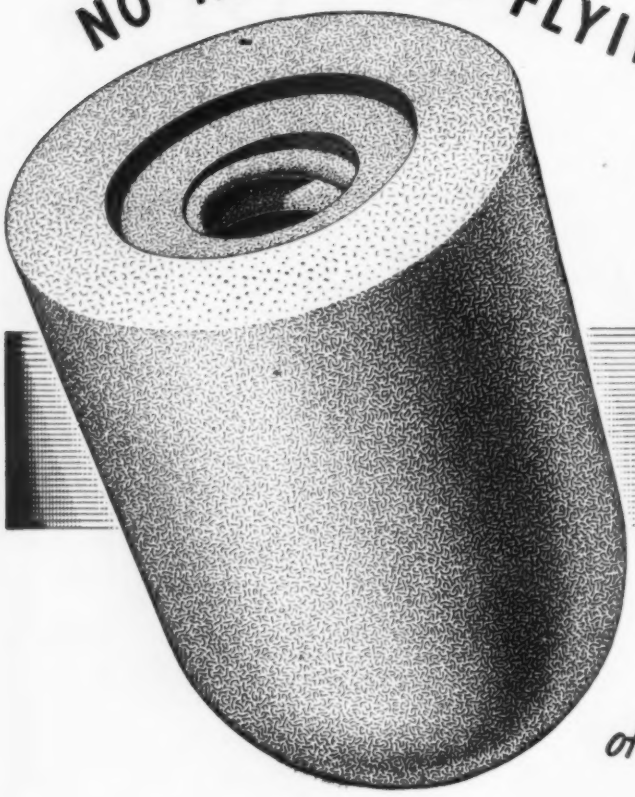
GWB

G.W.B. FURNACES LTD.

P.O. BOX 4, DIBDALE WORKS, DUDLEY, WORCS. TEL: DUDLEY 55455 (9 lines)
Associated with: Gibbons Bros. Ltd. and Wild-Barfield Electric Furnaces Ltd.

GWB218

NO NEED FOR FLYING STOPPERS!



The New, Tough

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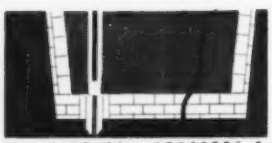
STOPPER END

offers **EXTRA** *Reliability*

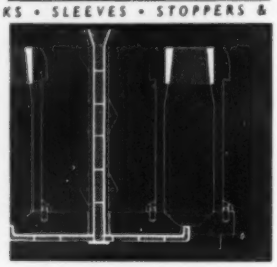
The focal point of teeming. The Stopper End. Subject to thermal shock, intense wear by liquid steel and severe mechanical stresses at high temperatures. No half measures with stoppers—they work or fail.

Resulting from years of development work Marshalls now present their "SR" quality. Already acclaimed and well-known in the biggest ladles of the major integrated plants, "SR" is equally satisfactory in the steel foundry and when teeming the smaller 'special' steel charges.

"SR" is highly refractory, accurately shaped—and tough. Consistently better and COST NO MORE.



LADLE BRICKS • SLEEVES • STOPPERS & NOZZLES • TRUMPETS & PIPES • CENTRE BRICKS & RUNNERS • MOULD HEAD BRICKS



MARSHALL

REFRACTORIES

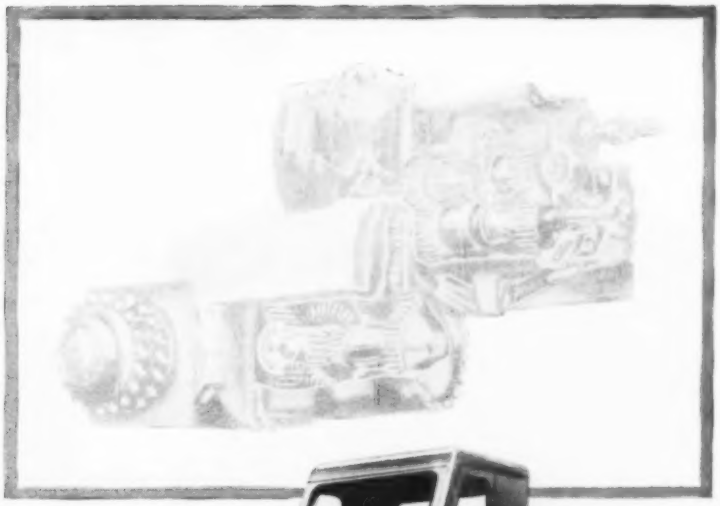
THOMAS MARSHALL & CO. (LOXLEY) LTD · LOXLEY · SHEFFIELD TEL: 343844/5/6

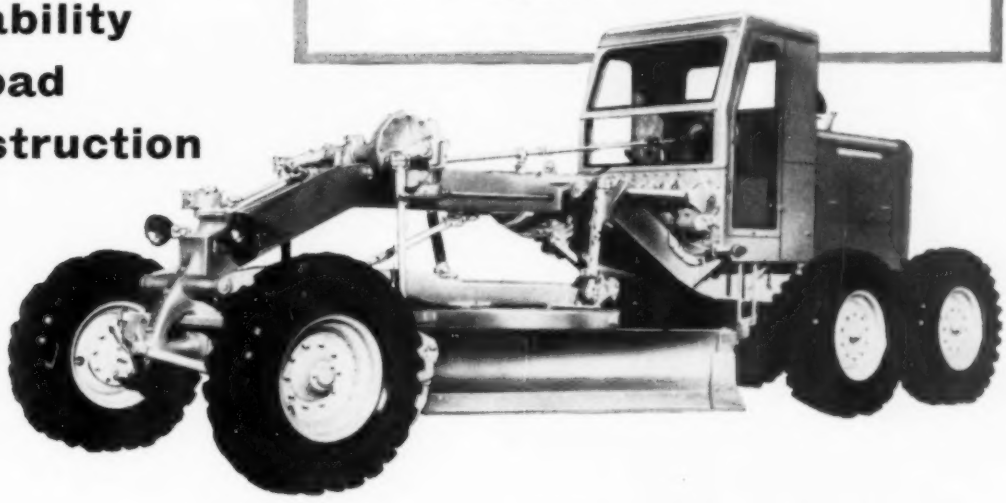
One of the MARSHALL REFRACTORIES GROUP of Companies

TM39

NICKEL ALLOY STEELS

ensure reliability in road construction





The power which operates and controls the Blaw Knox B.K. 12 Grader is transmitted through components of nickel alloy steels, notably the stub axles, track rods and half shafts, which are made from EN 25 2½ per cent nickel-chromium-molybdenum forged steel. This and other nickel alloy steels are used for such components as the main and subsidiary gears, pinions and pins which transmit the drive from the power unit and need good resistance to both fatigue and impact.

TYPICAL MECHANICAL PROPERTIES OF

EN25

These illustrate its strength and toughness in massive sections.

SIZE	HEAT TREATMENT	YIELD STRESS t.s.i.	MAXIMUM STRESS t.s.i.	ELONGATION per cent	IZOD ft. lb.
2½" dia.	Oil quenched 830° C. tempered 590° C.	75·2	78·8	17	44
2½" dia.	Oil quenched 830° C. tempered 650° C.	58·8	63·6	21·5	68
4" dia.	Oil quenched 830° C. tempered 650° C.	56·2	61·8	21	69
6" dia.	Oil quenched 830° C. tempered 650° C.	55·4	61·5	22	63

By utilising the better properties obtainable in more highly alloyed nickel alloy steels, dimensions can be reduced, lighter constructions produced, distortion through heat treatment minimised and reliability and economy achieved.

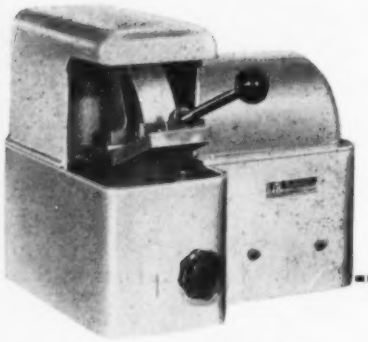
Send for 'The Mechanical Properties of Nickel Alloy Steels' ▶

TGA 21/1QA

MOND NICKEL

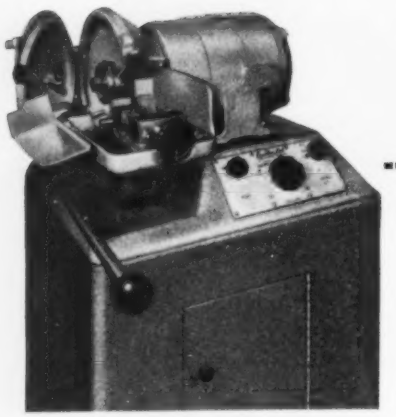
THE MOND NICKEL COMPANY LIMITED, THAMES HOUSE, MILLBANK, LONDON, SW1

3 SPECIMEN CUT-OFF MACHINES by Buehler



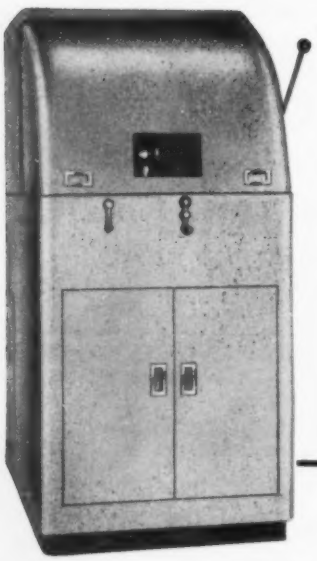
SENSITIVE CONTROL . . RAPID . . COOL . . CLEAN CUTTING

For Sections up to ½ in. The AB.1114 Specimen Cutter is ideal for the rapid, cool and clean cutting of small stock of ½ in. diameter. The specimen to be cut is fed into the wheel by the clamping vice lever thus enabling the operator to "feel" the cutting speed. Supplied either on pedestal or for mounting on bench, and equipped with coolant supply to both sides of abrasive wheel at point of contact with specimen.



For Sections up to 1 in. The AB.1010 Cut-Off Machine is of 1 in. diameter capacity, the coolant supply being fed from the inside of the wheel cover direct on to the abrasive wheel. The specimen to be cut is held securely in a vice fixed to the carriage which is fed into the wheel by the hand lever shown. The illustration shows only part of the cabinet which stands on the floor.

FULL PROTECTION FOR OPERATOR . . . . SENSITIVE CONTROL OF CUTTING FEED.



For Sections up to 3½ in. The AB.1000 is a fast cutting, sturdy Cut-Off Machine for dealing with specimens up to 3½ in. section. The cutting operation is effected by the hand lever Fig. 1 outside the cabinet body thus permitting the hood to be closed and protecting the operator against fragmentation and the effects of cutting toxic materials. Apart from the standard specimen clamp, a Subjet Attachment Fig. 2 is also supplied which enables the specimen to be completely submerged, high velocity jets of coolant also being sprayed on to both sides of the wheel immediately at the point of contact with the specimen.

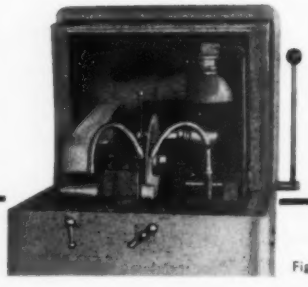


Fig. 1

SHANDON

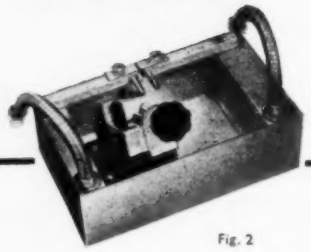


Fig. 2

SHANDON SCIENTIFIC CO. LTD., CROMWELL PLACE, LONDON, S.W.7.

Telephone: **KNIGHTSBRIDGE 1131.**

AUTOCARB

AUTOMATIC DEW POINT RECORDERS AND CONTROLLERS

Signal Controller

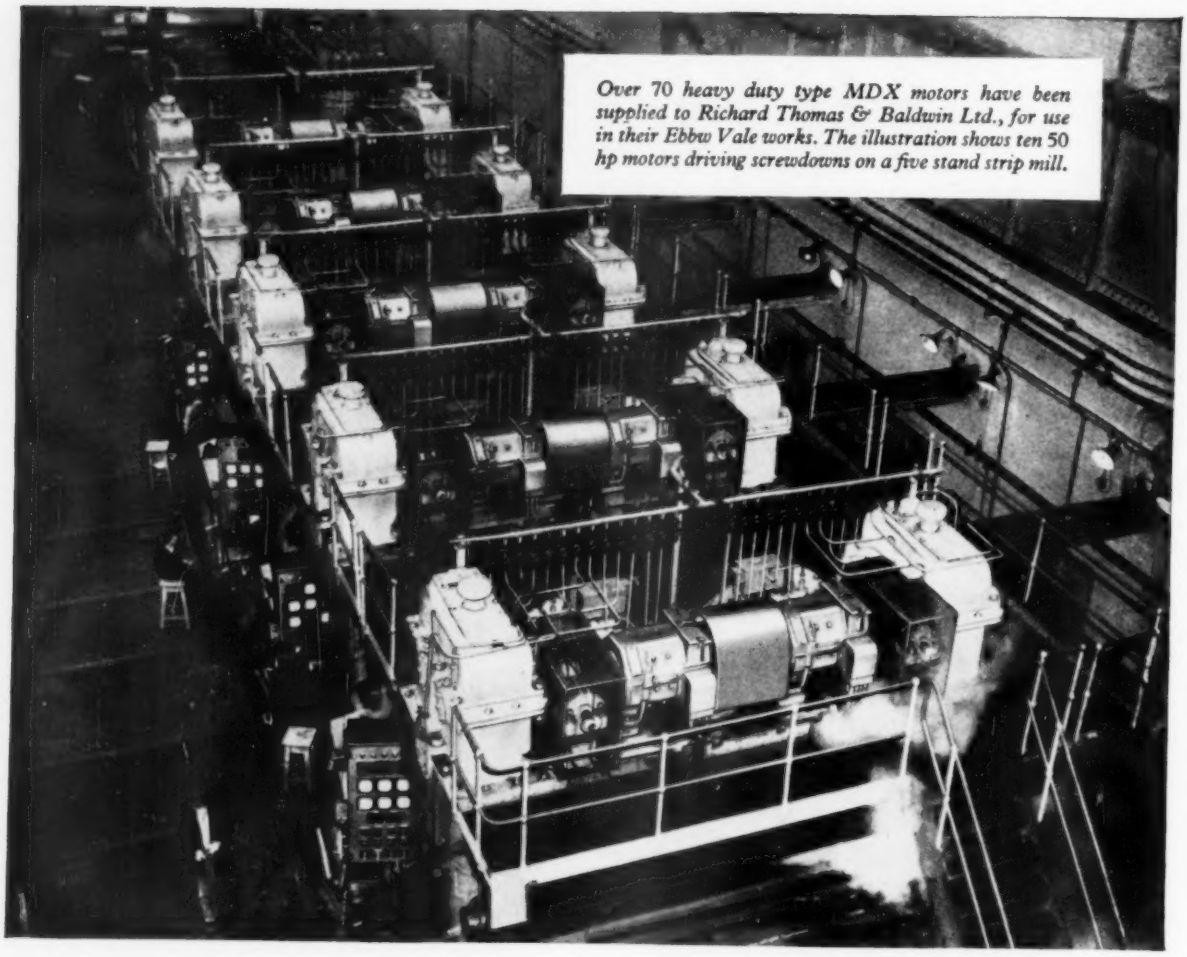
Recorder Controller

Autocarb systems automatically adjust the flow of air or enriching hydrocarbon to the furnace or generator in direct response to the dew point of the atmosphere. Specified conditions are automatically maintained. Autocarb systems are simple compact units which indicate, record and control the dew point of gas atmospheres in continuous or batch furnaces and atmosphere generators. They can control dew points to within plus or minus two degrees. On continuous furnaces separate zones of atmosphere control can be established and automatically maintained. If manual control is preferred, the Autocarb line includes simple recorders or indicators. On batch furnaces the Autocarb system will maintain the desired carbon potential for each temperature established in the heating chamber. On endothermic gas generators Autocarb systems automatically and continuously compensate for fluctuations in the composition of the reaction fuel gas supply and the humidity of the reaction air. This keeps constant the dew point of the product gas.

BRITISH FURNACES LTD.

CHESTERFIELD

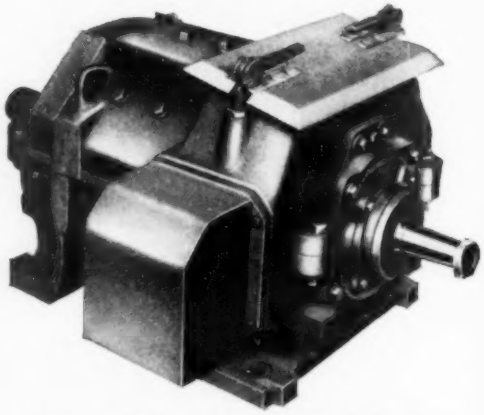
ASSOCIATED WITH SURFACE COMBUSTION CORPORATION, TOLEDO, U.S.A.



Over 70 heavy duty type MDX motors have been supplied to Richard Thomas & Baldwin Ltd., for use in their Ebbw Vale works. The illustration shows ten 50 hp motors driving screwdowns on a five stand strip mill.

All motors here are type MDX

A.E.I. type MDX motors have been specially designed to operate under the arduous conditions of iron and steel works. Their strength and reliability, and the exceptionally low inertia of the rotating parts make these machines ideal for driving a wide range of steelworks equipment. The use of mica and glass insulation suitable for high temperature operation enables the motor to withstand heavy overloads.
Available totally enclosed, up to 200 hp, or self-ventilated, up to 250 hp.



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THE NEW **PHILIPS** *EM 200*

10 Å RESOLVING POWER & BETTER

...with traditional **PHILIPS** *ease of control*

The new EM 200 electron microscope stems from the Philips experience and research that produced the EM 100 and EM 75. Though it has a high performance, it is remarkably easy to use — a most important feature in this type of equipment.

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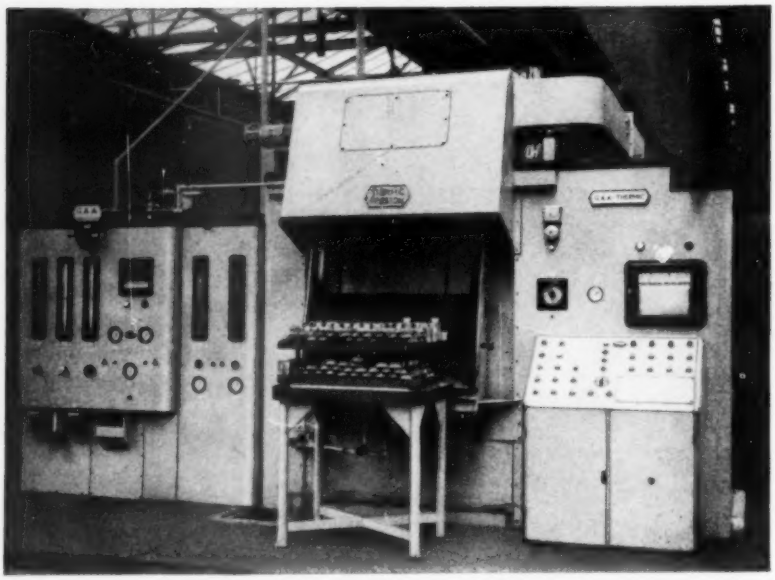
(PCL 0434)

Heat Treatment
–Gas Board Demonstrates latest developments.

IN this modern general metals age of ours it seems a far cry to the days when the heat treatment of metals was considered to be a 'black art' and when the closely guarded secrets of the swordmakers and workers in precious metals were handed down from father to son. Today we recognise the value of the comparatively new technology of metallurgy and realise the benefits our civilization receives from the new metallic alloys and from the new plants and methods of processing these alloys which have been developed during the last few decades.

The type of plant we have in mind includes such items as the new heat treatment furnace which the West Midlands Gas Board recently has had installed in its Heat Treatment Service establishment located at Adderley Street, Birmingham 9. This establishment was brought into being by the Birmingham Gas Department more than 40 years ago and the plant has been continually brought up to date. The experience of the metallurgical staff of the 'service' is considerable and this is reflected in the quality of the supervision given to all the heat treatment processes carried out at Adderley Street, as well as in the advice given to consumers concerning the use of gas fired plant. This new item of plant, one of the first of its kind to be installed in the Midlands, is an atmosphere controlled batch-type unit, capable of accepting a total load measuring 3 ft. 0 in. wide, 2 ft. 0 in. deep and 1 ft. 6 in. high, and operating up to temperatures of 950° C. The furnace itself comprises two separate chambers, a hot zone, heated by recuperative gas fired nickel-chrome radiant tubes, placed above and below the work chamber; and a water jacketed discharge vestibule. An oil quench tank is incorporated below this vestibule and both the vestibule door and the inner furnace door are power operated.

Controlled atmospheres, suitable for a wide variety of treatments including clean hardening and annealing, gas carburising and carbonitriding, are supplied from an endothermic generator forming part of the plant. This generator is fired by towns gas but uses propane gas and air for the production of the endothermic gas. Both propane and ammonia enrichment are provided, for use when gas carburising or carbonitriding, and it is possible to vary the 'dew point' of the atmosphere gas in order to provide clean heat treatment of steels over a wide range of specifications.



This Gas-fired furnace of new design for gas carburising and clean heat treatment in controlled atmospheres is a working exhibit at the Board's Heat Treatment Service establishment.

The furnace is operated from a control panel and desk which provides automatic furnace temperature recording control, atmosphere control and push button control of all the power operations attendant upon the charging, discharging and quenching of the furnace load, which thus become automatic operations, each action being indicated by signal lights on the panel. Vertical flow gauges and dial gauges incorporated in the panel complete the instrumentation of the plant, and the panel thus gives the operator an immediate picture of the stage of the process and the state of the plant at any given time.

In order fully to demonstrate this plant to prospective purchasers it is of course necessary to operate it on a commercial heat treatment shop basis and the Board's Heat Treatment Service is open to receive orders for the gas carburising of such items as crown wheels and pinions, gears, cams and cycle components, clean annealing, hardening, and tempering of special die blocks, machined parts and similar items. All this work would be carried out to the requirements of the client concerned, would be supervised by qualified metallurgists, and when necessary would conform to the dictates of any inspection approval.

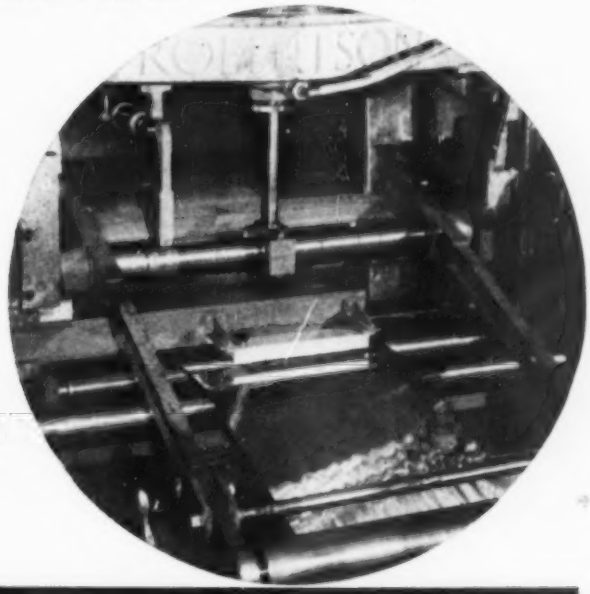
Industrialists, metallurgists and plant engineers are invited to inspect any of the items of plant at Adderley Street either with a view to installing similar plant at their own works or for the purpose of assessing the usefulness to their own organization of the commercial heat treatment facilities provided by the 'Service'.

This is an advertisement by the West Midlands Gas Board

Baldwin Atomat Nucleonic Thickness Gauges

ACCURATE, CONTINUOUS, NON-CONTACT INSPECTION

SAVES MATERIAL, LABOUR AND TIME!



The Atomat Principle

RAY SOURCE

MATERIAL

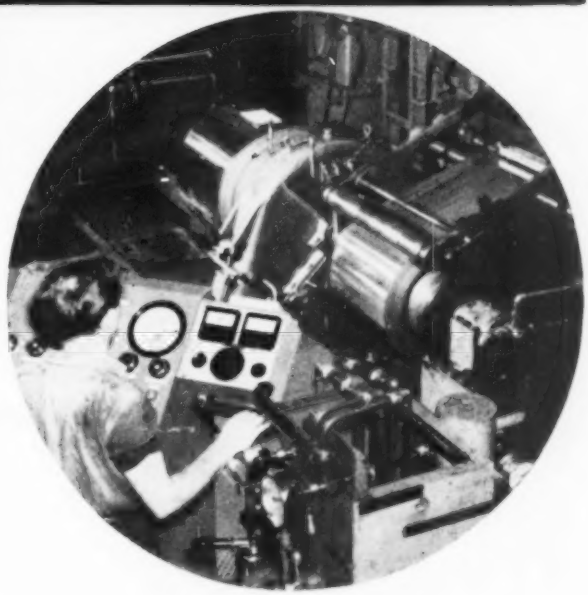
RAY DETECTOR

Amount of rays passing through material indicates thickness or weight per unit area.

That's the very basic explanation of the Atomat principle. There are three main gauges; the Atomat Cold Strip Gauge, the Hot Strip Gauge and the Beta Gauge.
In addition there are special purpose variations, made to meet the requirements of many different industries.

HERE'S WHAT AN ATOMAT GAUGE CAN OFFER YOU

- **Time and material saving both in production and setting up**
- **High accuracy**
- **Calibration in your usual units of measurement**
- **Continuous readings for materials in constant production**
- **Auto standardisation and control if you require**
- **Minimum maintenance**



Baldwin Atomats are already boosting production and cutting costs in the paper, metal rolling, rubber and plastics industries. Highly successful applications also include the measuring of selenium on aluminium, coatings of paint and lacquers, density of wood chips, coatings of grit on abrasive papers and padding and resin impregnation in the textile industry. But the potential of Baldwin Atomat Nucleonic Gauges has hardly been tapped.
Is there a place for them in your plant?

INSTRUMENT DIVISION

Send for the Baldwin Brochure MA 124. It gives full information on Atomat Gauges clearly and concisely.

BALDWIN INDUSTRIAL CONTROLS

Baldwin Instrument Company Limited, Dartford, Kent A Harper Group Company

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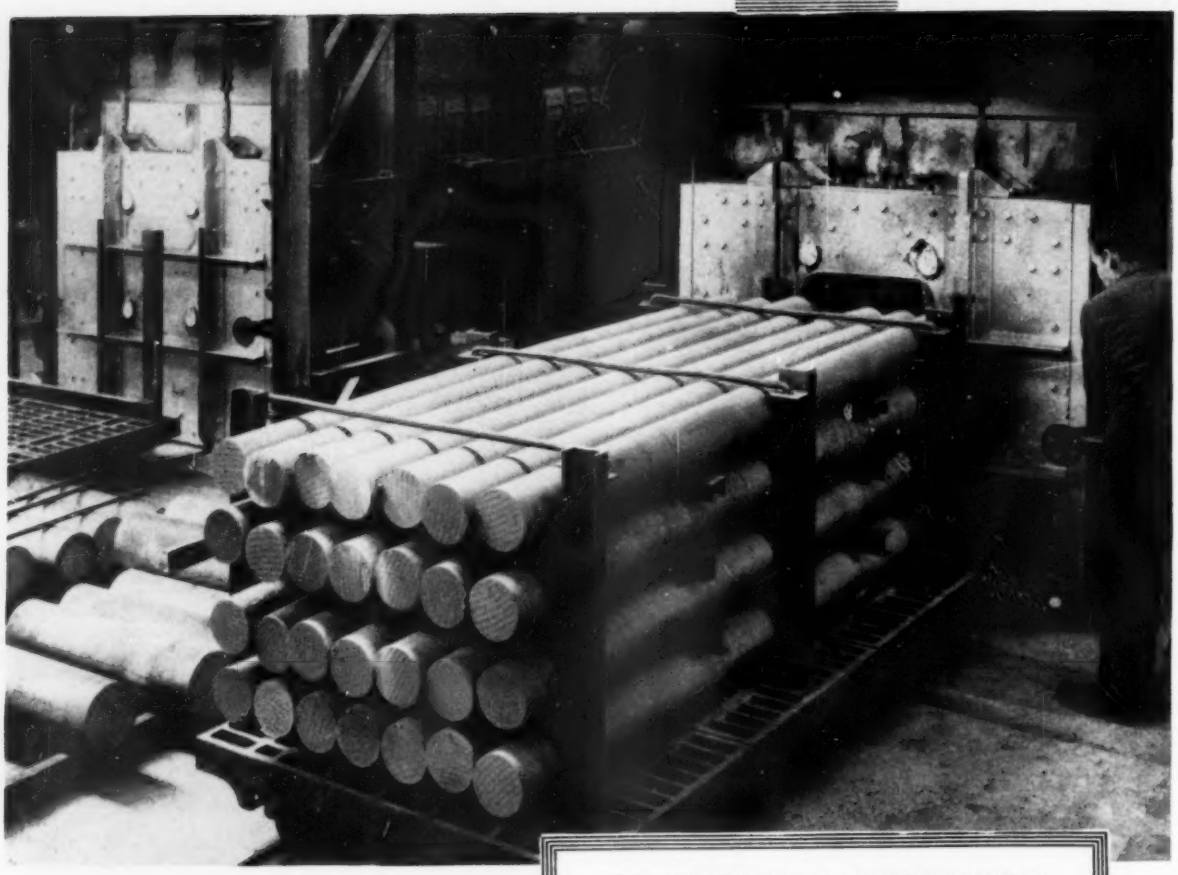

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Messrs. Reynolds T. I. Aluminium Co. Ltd.

TWO DIRECT GAS-FIRED CONVECTION TYPE FURNACES WITH HIGH SPEED RECIRCULATING FANS FOR HOMOGENISING ALUMINIUM BILLETS

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GIBBONS BROTHERS LTD

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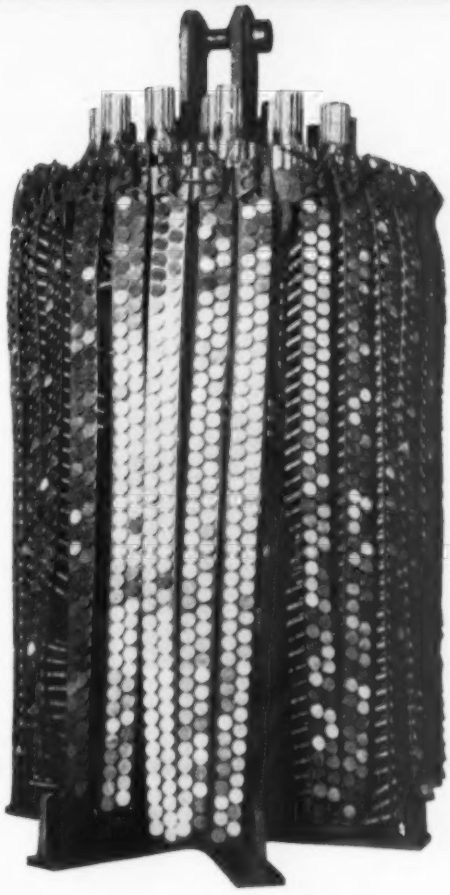
TELEGRAMS: "GIBBONS, DUDLEY" TELEPHONE: DUDLEY 55141 (P.B.X.)

Economy pack



Compactness of furnace layout is matched by the amazing density of loading in these INCANDESCENT gas carburizing furnaces. Such performance is made possible by two special INCANDESCENT features

Circulating fan which ensures consistent uniformity of case depth.

Jetube heating with its special recirculation system which transmits heat to the load with maximum speed and efficiency.

Photographs by courtesy of The Ford Motor Co. Ltd.

INCANDESCENT

THE INCANDESCENT HEAT CO. LTD. · SMETHWICK · ENGLAND

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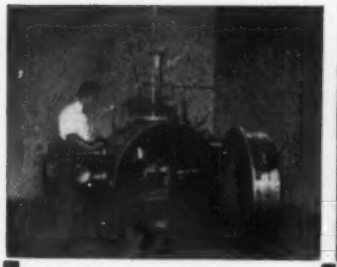
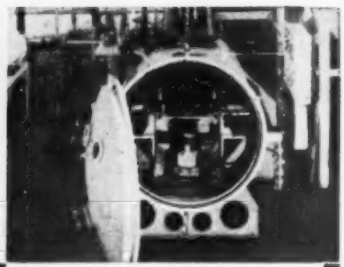
. . . COULD A VACUUM FURNACE DO THE SAME FOR YOUR PRODUCTS?



Model 2555 Vacuum Induction Furnace with melting capacity of 50 pounds of steel. Other standard furnaces have capacities of 12 to 3,000 pounds.



Model 2705 Non-Consumable Arc Skull Furnace with a capacity of 50 pounds of titanium. Other standard vacuum arc furnaces have capacities of 8 to 10,000 pounds of titanium.

ELECTRIC WILD BARFIELD FURNACES

for all heat-treatment purposes

Metals for parts to withstand jet engine temperatures and stresses can be produced in vacuum furnaces. Such metals enable components to be made with fewer rejects. If your products need parts that operate under severe conditions or need high purity metals or alloys—a vacuum furnace will help you to produce them.

Wild-Barfield can supply equipment made to the designs of and tested by the unrivalled experience of the National Research Corporation who have built and operated more high vacuum furnaces than any other company in the world.

Write for details of the Wild-Barfield—NRC range of high vacuum plant.

WILD-BARFIELD—NRC EQUIPMENT INCLUDES:

VACUUM INDUCTION FURNACES
VACUUM ARC FURNACES
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VACUUM ANALYSING EQUIPMENT
HIGH VACUUM DIFFUSION PUMPS
LABORATORY VACUUM FURNACES

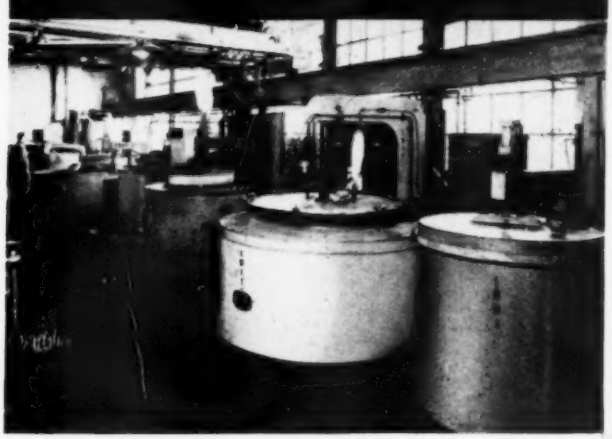
NRC *is the trade-mark of the National Research Corporation, registered in the United States Patent Office.*

WILD-BARFIELD ELECTRIC FURNACES LIMITED

ELECFURN WORKS · OTTERSPOOL WAY · WATFORD BY-PASS · WATFORD · HERTS. Telephone: Watford 26091 (8 lines)

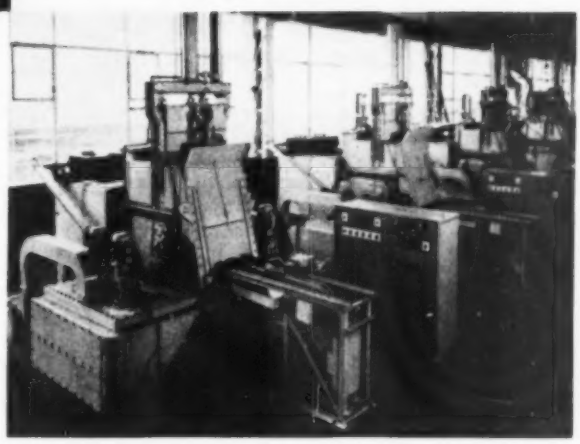
WB 84

the choice of the motor manufacturer . . .



An installation of pit type furnaces with separate cooling chambers and oil and water quench tanks for carburising and hardening motor gear box parts.

EFCO furnaces



Humber Ltd chose Efco-Lindberg horizontal closed quench furnaces for carburising and carbonitriding gear components. The furnaces are electrically heated with Corrtherm elements.

Austin, Ford, Humber, and other leading motor manufacturers chose Efco Furnaces for hardening, gas carburising or carbonitriding gear components.

When you decide on Efco furnaces you may pick from the best of the World's current designs. For carburising and carbonitriding there are both vertical and horizontal closed quench furnaces, and vertical furnaces with separate cooling chambers. For hardening there are many continuous designs including shaker hearth, roller hearth and pusher furnaces.

EFCO

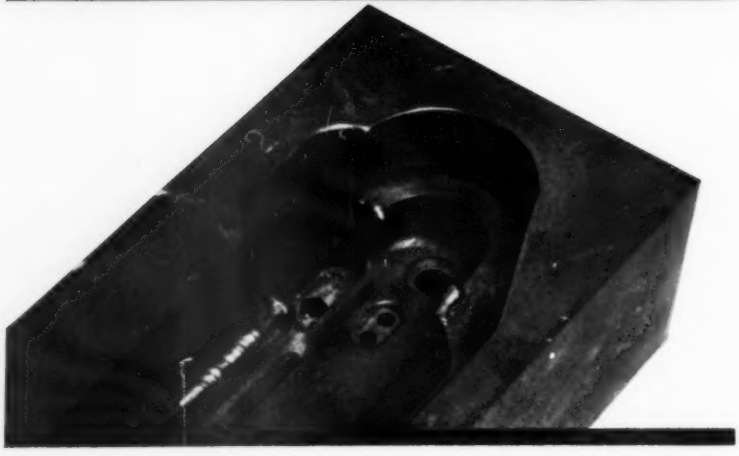
BEST OF THE WORLDS CURRENT FURNACE DESIGNS

ELECTRIC RESISTANCE FURNACE CO. LTD.

NETHERBY, QUEENS ROAD, WEYBRIDGE, SURREY · Weybridge 3891.

Associated with Electro-Chemical Engineering Co Ltd

for repairing damaged or worn tools



STAG

ATHYWELD

DEPOSIT WELDING PROCESS

Most intricate repairs or welds can be accomplished by this process on high alloy steels, mainly because the process enables a delicate control to be kept metallurgically and physically. The process eliminates fierce heating and cooling of the parts.

Damaged alloy steel dies for pressure castings, etc. can also be repaired. Above is a group of H.S.S. cutters and alloy dies as received by the Engineers' Tool Department before welding, the arrows indicating a few of the areas of damage, perfectly restored after welding.

The close-ups show the damage to important faces of a die, and the same die after welding before finish machining.

Edgar Allen & Co. Limited

IMPERIAL STEEL WORKS · SHEFFIELD · 9

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SHEFFIELD 9

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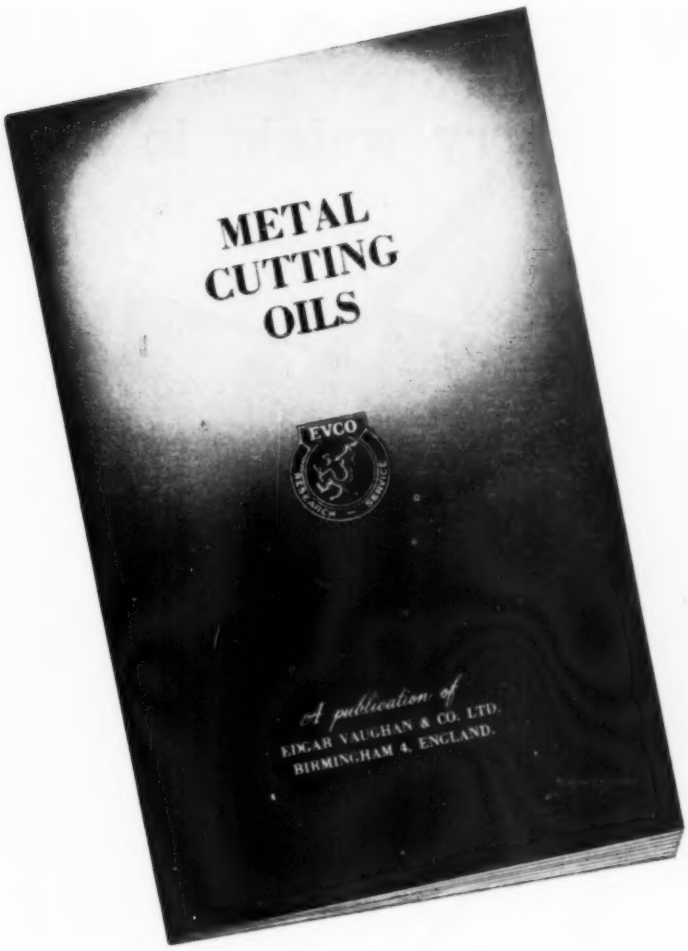

A copy for the asking . . .

In days when time is money and competition gets keener, the importance of cutting oils and lubricants is often overlooked.

To help engineers and others concerned with the selection of the *right* cutting oil for a particular operation, we have produced the above booklet. If you have not received a copy, please—in your own interest—write for one to-day, on your business heading.

The booklet is not a general treatise on cutting oils. It is, nevertheless, a helpful guide in the selection—with the advice of Edgar Vaughan & Co. Ltd.—of the most suitable and economical oil or lubricant for a specific job of work.

In a general way, the booklet covers Neat Cutting Oils, Broaching Oils, Soluble Oils of all types, Grinding and Drawing Compounds.

Edgar
Vaughan
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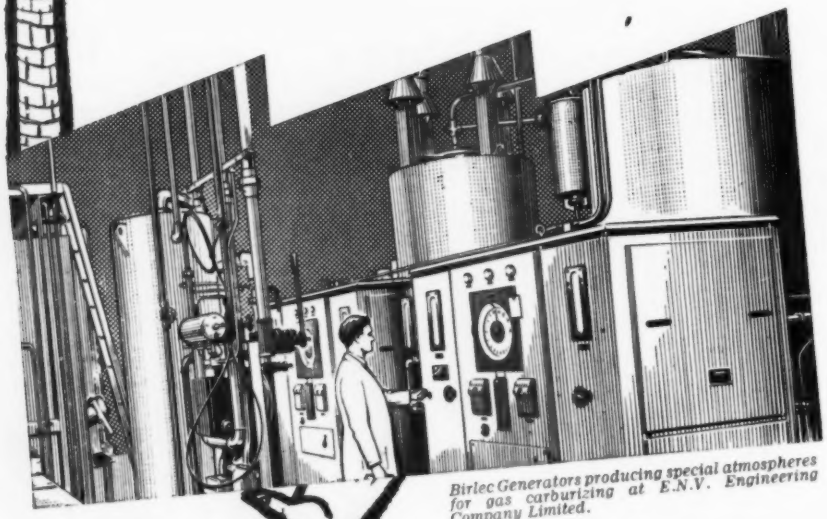
Works and depots at: Birmingham, Manchester, Liverpool, Southall (Middx.), Bristol, Glasgow.

SHELL-MEX AND B.P. GASES

carry weight in industry



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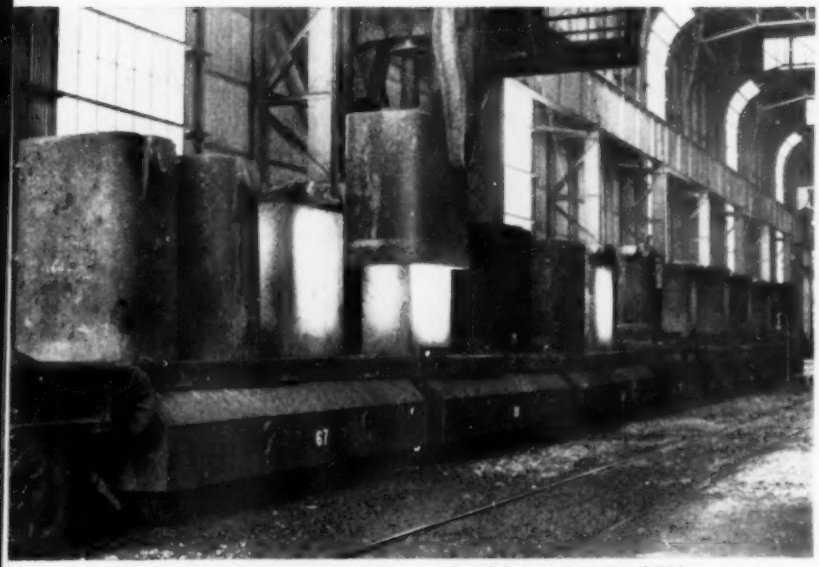

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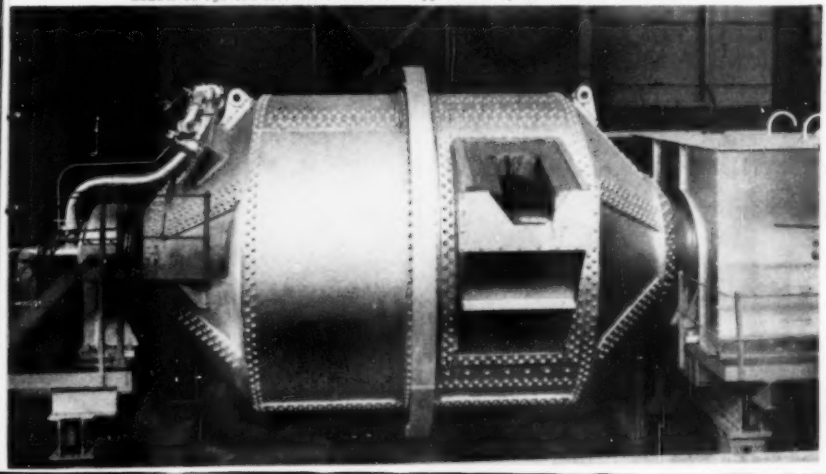

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METALLURGIA

THE BRITISH JOURNAL OF METALS
INCORPORATING THE METALLURGICAL ENGINEER

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METALLURGIA

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INCORPORATING THE "METALLURGICAL ENGINEER"

November, 1959 Vol. LX. No. 361

Progress in Steelmaking

THE news that the melting shops of the Steel, Peech and Tozer branch of The United Steel Companies are to be transformed, the open hearth furnaces being completely replaced by electric furnaces, gives point to an article entitled "Revolution in Steelmaking," which appeared in the October issue of *Steel Review*, in which the author points out that the three principal methods of making steel in bulk—the acid Bessemer, the basic Bessemer, and the open hearth—were all invented between 1856 and 1878, and were responsible for making practically all the commercial steels until the First World War. Since that time the electric furnace, both arc and induction types, has made great strides. One of the principal advantages of electric furnace melting is the ability to operate on 100% scrap charges and to produce steel of high quality. On the other hand, the conversion costs are higher than in the traditional steelmaking processes, and until recently the process has largely been confined to the production of steels of special high quality. The development of larger furnaces, however, has led, particularly in the United States, to its use for the manufacture of other steels. The largest arc furnaces in operation in this country at present are the two 60-80 ton units at the Stocksbridge works of Samuel Fox & Co., Ltd., but the six units to be installed in Steel, Peech & Tozer's Templeborough melting shop will each have a capacity of 110 tons. Although, when completed, Templeborough will be the largest all-electric melting shop in the country, it will not be the only one, as there is already in existence the Brymbo plant where the open hearth furnaces have been dismantled and replaced by 40 ton arc furnaces. Brymbo also has the distinction of being the first electric furnace plant operating on scrap and pre-refined pig iron.

We take this opportunity of extending to Readers our Cordial Greetings for the Festive Season and Good Wishes for the Coming Year.

Although electric steelmaking is undoubtedly on the increase, it is likely that, for some time to come, the bulk of British steel will be produced by the Bessemer and open hearth processes, or modifications of them. Historically the acid Bessemer was the first process for bulk steel production, followed by the open hearth process, which could handle scrap, and later by the basic Bessemer process. The latter is known on the Continent—where it is widely used—as the Thomas process, after Sidney Gilchrist Thomas, who invented it in this country in 1878. The process adopted by any company depends on a number of factors, both technical and economic. A Bessemer plant involves lower capital expenditure at the steelmaking stage, and has lower operating costs than the open hearth, but it cannot deal with large amounts of scrap and it is more demanding in its raw material requirements. Furthermore its product, particularly by the basic process, is not universally acceptable. In fact, owing to difficulties arising from the high sulphur and alumina content of some of the home ores, and the consequent variability of the pig iron made from them, the basic Bessemer steel which was the final product could not be relied upon to give the quality demanded by the great steel-consuming industries. In consequence, British Standard Specifications ruled out basic Bessemer steel for many purposes, and the process in this country fell into disuse in the early 1920's. Throughout the years, however, the acid Bessemer plant at Workington has maintained its reputation for the manufacture of steel rails by virtue of the low sulphur and phosphorus content of the Cumberland hematite ore on which it is based.

The 1930's saw the rebirth of the basic Bessemer process, when Stewarts and Lloyds set up a new plant at Corby, where, by preparation of the local Northamptonshire ores and treatment of the pig iron with soda to deal with the sulphur, it was proved to be feasible and economic to make dead soft steel for tube-making by the basic Bessemer process. A similar development was made in 1938 for making dead soft sheet steel by this process at Ebbw Vale. To put these developments into perspective, however, it should be pointed out that at the end of the war, the open hearth process accounted for 88% of the steel produced in this country, the remaining 12% being equally divided between the Bessemer and electric processes.

Reference was made earlier to the drawbacks to the Bessemer process. Two of these—the high phosphorus content required in the pig iron and the high nitrogen content of the steel produced—have their origin in the fact that the liquid iron is blown with air. If the metal were blown with oxygen instead of air, there would be no nitrogen pick-up and such a high phosphorus content would not be needed to maintain the required temperature in the melt, as the heat losses in the form of hot nitrogen waste gas would be eliminated. With the development of tonnage oxygen plants, the basic Bessemer method has been modified in two ways in Germany. In some cases the nitrogen content is reduced by replacing part of the blowing air by oxygen, and as an alternative the air blast is replaced entirely by a mixture of roughly equal proportions of oxygen and steam. In the latter case the resulting steel is almost free of nitrogen. Both the Corby and Ebbw Vale plants are equipped for blowing with either air or oxygen/steam, according to quality, and the new Bessemer plant recently installed at the Abbey Works of the Steel Company of Wales is entirely oxygen/steam blown for the making of special dead soft steels.

As a result of experiments in Austria since the Second World War, what is known as the L-D process has been developed, in which the air blast of the normal

converter is replaced by a jet of tonnage oxygen directed on the surface of the liquid pig iron, and travelling at supersonic speed. The oxygen penetrates into the metal and carries out the conversion into steel at almost the same rate as in the traditional process. This process combines the advantages of low capital expenditure in the steel plant, high speed of working and low operating costs, with a wider application range as regards phosphorus content, and gives steel with a very low nitrogen content. After prolonged testing, this steel is now accepted by Lloyds inspectors as equivalent to open hearth steel in many specifications. This, of course, means that it is possible to produce open hearth quality steels at Bessemer prices. The L-D process is to be the sole means of steelmaking at the new Colville and Richard Thomas & Baldwin strip mill plants.

Oxygen blowing has also been developed for normal electric arc furnaces, where marked economies can be effected in the manufacture of alloy steels, particularly of the stainless type. At the Brymbo plant referred to earlier, the key unit is the specially designed tiling furnace in which oxygen and lime powder are blown into the liquid pig iron and most of the impurities are removed before the metal is transferred to the 40-ton arc furnaces for final refining.

In the open hearth process, too, oxygen is playing an increasingly important role, with a view to increasing the rate of working and reducing the fuel consumption. The metallurgical load on the furnaces, which is higher in Britain than elsewhere, is reduced in some plants by blowing the pig iron with oxygen in a ladle before charging it to the open hearth furnace. Oxygen lancing may also be used in the open hearth furnace itself to accelerate the rate at which carbon is removed, at the same time reducing the operating time and lowering the fuel consumption. The latest development in open hearth practice is to apply the same principles as in the L-D converter. As soon as the charge is partially melted, a jet of supersonic oxygen is applied above the surface of the liquid metal. This penetrates into the metal, oxidises the impurities at a very high rate, and generates so much heat that a cooling agent—either scrap or ore—must be added : no external fuel is required during this period. This new application has been developed in the large Ajax tilting furnaces at Appleby-Frodingham, and in a number of fixed open hearth furnaces at other works.

It seems likely that there will be an increasing use of these oxygen-blowing and oxygen-enrichment developments in this country, particularly for steels made from charges containing a high proportion of pig iron, leaving the melting of scrap to open hearth furnaces and electric furnaces. The rate of steel production will be considerably increased, and it is expected that there will be a corresponding reduction in costs, so that, without sacrificing quality, the British steel industry will be in a strong position to meet the competition of the new processes in world markets.

Meeting Diary

4th January

Institution of Works Managers. Open Discussion on Management Problems with SIR ALEXANDER FLECK. Board Room of the Manchester Chamber of Commerce (rear entrance), King Street, Manchester. 6.30 p.m. Admission by ticket only, obtainable from S. S. Brown, 463, Kings Road, Stretford, Manchester.

5th January

Institute of Metals, Oxford Local Section. " Brittle Fracture ", by PROF. N. J. PETCH. Cadena Cafe, Cornmarket Street, Oxford. 7 p.m.

Institute of Welding. Christmas Lecture to Young People : " The Magic Arc " by PROF. H. O'NEILL, 54, Princes Gate, London, S.W.7. Ticket only.

Sheffield Metallurgical Association. Annual General Meeting of the Methods of Analysis Group and Presidential Address by the New Chairman. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield, 3. 7 p.m.

6th January

Institute of Welding, Manchester and District Branch. " Inspection and Interpretation of Welding Codes ", by G. ATKINSON. Reynolds Hall, Manchester College of Science and Technology. 7.15 p.m.

7th January

Institute of Metals, London Local Section. " The Rolling of Aluminium ", by DR. F. KASZ. 17, Belgrave Square, London, S.W.1. 6.30 p.m.

Leeds Metallurgical Society. " Modern Developments in Metallurgical Analysis ", by K. M. BILLS.

12th January

Institute of Metals, South Wales Local Section. " Metals in Antiquity ", by L. BIEK. Royal Institution, Swansea. 7 p.m.

Institution of Plant Engineers, Manchester Branch. Paper by PROF. F. C. WILLIAMS, O.B.E., F.R.S. Engineers' Club, Albert Square, Manchester. 7.15 p.m.

Sheffield Metallurgical Association. Annual General Meeting of the Refractories Group and Presidential Address by the New Chairman. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield, 3. 7.30 p.m.

13th January

Institute of British Foundrymen, Southampton Section. Technical Film Show : " The Foundryman's Raw Materials ". Liaison meeting with guest members from London. Technical College, St. Mary's Street, Southampton. 7.30 p.m.

Institute of Welding, South London Branch. " High Temperature Brazing—A Modern Joining Method ", by E. R. PERRY. 54, Princes Gate, London, S.W.7. 7.30 p.m.

Manchester Metallurgical Society. " The Newer Methods of Steelmaking ", by J. PEARS. Manchester Room of the Central Library, Manchester. 6.30 p.m.

Society of Chemical Industry, Corrosion Group. " Corrosion Problems of High Temperature Pressurised Water Reactors ", by J. N. WANKLYN. 14, Belgrave Square, London, S.W.1. 6 p.m.

14th January

Institute of British Foundrymen, Beds and Herts. Section. " Properties of Copper-base Alloy Castings ", by F. HUDSON. Town Hall, Luton. 7.30 p.m.

15th January

West of Scotland Iron and Steel Institute. Joint Meeting with the **Iron and Steel Institute, Engineers' Group.** " Engineering Aspects of a Modern Open-Hearth Steel Plant ", by J. A. KILBY and J. C. GORDON. 39, Elmbank Crescent, Glasgow. 6.45 p.m.

16th January

Non-Destructive Testing Society of Great Britain. " The N.D.T. of Magnesium Alloys ", by F. L. ALDRIDGE. Engineering Centre, Stephenson Place, Birmingham. 10 a.m.

18th January

Institute of British Foundrymen, East Anglian Section. " Production of Foundry Sands ", by A. R. ABRAHAM. Liaison meeting with guest members from London. Lecture Hall, Public Library, Ipswich. 7.30 p.m.

Institute of Physics, Manchester and District Branch. " Recent Developments in Scintillation Counting ", by DR. J. F. BIRKS. Bragg Lecture Theatre, University of Manchester. 7 p.m.

The Use of Malonic Acid as an Anodising Electrolyte

By J. M. Kape,* M.Sc.Tech., A.R.I.C., A.I.M.

Malonic acid used as an anodising electrolyte is capable of producing on a wide variety of alloys coatings having properties notably superior to those produced by conventional anodising methods. The author discusses the various anodic films which can be obtained from a 12·5% w/v electrolyte using different current densities, temperatures and voltages. The coatings formed are of a deep yellow or sepia colour, and can be extremely hard, corrosion resistant and abrasion resistant, and possess a high inherent light fastness.

DURING work on the anodising of aluminium and its alloys, carried out in the Research Division of High Duty Alloys, Ltd., it was noted that a cold dilute solution of malonic acid in water functioned rather as a barrier-forming electrolyte, insofar as when a potential difference was applied to an aluminium anode, the current density rapidly fell to nearly zero at any applied potential between 0 and 120 volts. The literature on the use of malonic acid as an anodising solution for aluminium alloys was very scanty, and revealed that malonic acid was accepted to cause " valve action " of aluminium,[1] and two early patents implied that it was of use in a mixed organic electrolyte to form opaque white films,[2] and mixed with sulphuric acid to facilitate the production of hard coatings.[3] There appeared to be no reference describing the use of straight malonic acid solutions as anodising electrolytes, and the work to be described was therefore undertaken.

EXPERIMENTAL

Early Work[4]

Initially, various concentrations of malonic acid crystals were dissolved in water in stainless steel containers which functioned as cathodes, and it was discovered that the more dilute the solution the higher the voltage required to pass any sizeable current density at any one temperature. Ranges of concentration of electrolyte from 3 to 25% were employed, and it was soon discovered that, in order to use malonic acid in accepted anodising practice—namely to pass a certain current (say 12–15 A./sq. ft. of anode surface) for any length of time and obtain a uniform coating—it was necessary to :—

(*a*) Use a moderately concentrated electrolyte (10–15% w/v).

(*b*) Use a warm-hot solution (40–60° C.).

(*c*) Employ a means of very vigorously agitating the electrolyte.

Although anodic films could be obtained in cold dilute malonic acid solutions, they were very thin (below 0·0001 in.) and obviously very non-porous with a thick barrier layer, as the operating voltage was often 120 (the maximum obtainable with the generating equipment in use).

Vigorous agitation was essential if coatings having a uniform appearance were to be produced, as in a stagnant electrolyte during anodising it appeared that a viscous, wedge-shaped film of electrolyte was flowing up the sides of a flat panel. This film of electrolyte was thick at the top of the panel and thin at the bottom and, presumably, allowed the ions responsible for anodic oxidation to penetrate it at the base of the panel, but not at the top. Hence, the finally anodised panel, when checked for film thickness with an isometer, would appear thick at the bottom end and become progressively thinner towards the top end. Correspondingly, the yellow colour of the anodic coating was darker at the base of the panel.

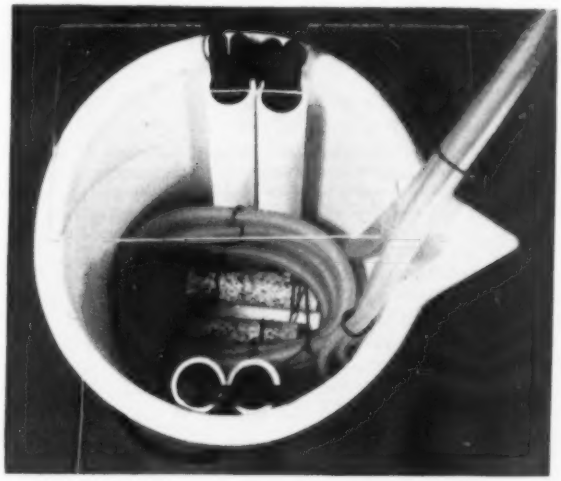

Fig. 1.—General view of the anodising equipment.

Stainless steel as a cathode metal was not entirely satisfactory, using malonic acid solutions, particularly at temperatures much greater than 60° C., when vigorous dissolution of the stainless steel occurred. Moreover, at such temperatures, some decomposition of malonic acid to acetic acid occurred, with a corresponding acceleration of the attack on the steel.

As a result of these investigations, it was decided to investigate fully the anodising characteristics of a 12·5% w/v vigorously agitated malonic acid electrolyte contained in a plastic beaker and using inert carbon cathodes.

* Formerly in charge of metal finishing research at High Duty Alloys Ltd., now general manager of B. T. Anodising Co., Ltd.

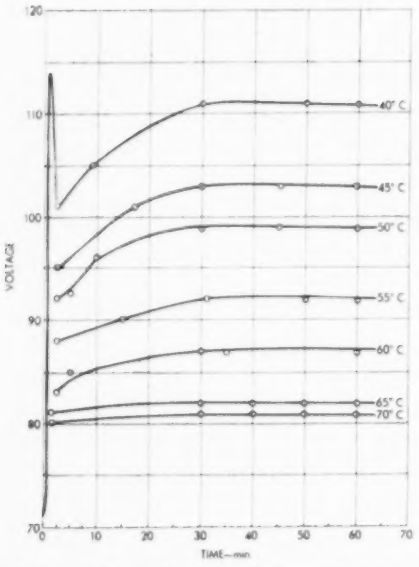

Fig. 2.—Variation of voltage with anodising time at various bath temperatures: malonic acid (12·5% w/v); current density 12 A./sq. ft.

The 12·5% w/v Electrolyte

The apparatus is shown in Fig. 1, and consists of a 2 gallon polythene bucket fitted internally with small porous porcelain air bubblers and polythene cooling coil. All tied connections were made with P.V.C. thread. The carbon cathodes consisted of thin carbon arc electrode rods, surrounded by cylindrical porous pots, closed at one end in order to prevent flaking of carbon into the electrolyte.

The first investigation was concerned with the range of operation of the electrolyte, with respect to both temperature and voltage relative to the current density applied. The material employed was 1C alloy (99·5% Al) containing 0·4% Fe, 0·2% Si and about 0·03% Cu.

Panels, 2 in. × 3 in. × 16 s.w.g., were etched in a 10% w/v solution of caustic soda at 55–60° C. for 5 minutes, dipped in nitric acid, dried and weighed. They were anodised in malonic acid for various times at various temperatures and various current densities, rinsed for 3 minutes in running water, and weighed after drying. They were then sealed for 30 minutes in boiling distilled water, at a *p*H of 5·5, and dried and weighed. Stripping of the anodic film was accomplished by immersion in a solution containing 35 ml. phosphoric acid (s.g. 1·75) and 20 g. chromic acid in 1 litre of distilled water, used at the boiling point, until no anodic coating remained on the surface. After drying, the panels were weighed.

Figs. 2 and 3 show a plot of anodising voltage against time of anodising at various temperatures, using current densities of 12 and 24 A./sq. ft. of anode surface, respectively. Fig. 2 indicates the typical type of voltage-time relationship in full, for the 40° C. curve; there was not room to indicate this clearly for all the curves. It is seen that, initially, in order to maintain the current density, it is necessary to raise the voltage rapidly during the first minute—this action presumably forming the barrier layer—in accordance with work on other electrolytes by Hunter and Fowle.[4] During the next few seconds, the barrier layer perforates, forming pores and allowing the electrolyte to penetrate to the base metal, and the film commences to grow. The perforation of the barrier layer causes the current to begin to rise and, in order to maintain a constant current density, the voltage must be reduced to a value known as the forming or starting voltage. Once this has been done, the voltage must be raised slightly over a period of time to maintain the current density constant as the anodic film grows.

Examination of Figs. 2 and 3 also reveals that the forming voltage is lowered by increase of temperature, and is, in fact, extremely sensitive to changes of temperature as small as 0·5° C. This is illustrated in Fig. 4, which shows the relationship of forming voltage to temperature at three different current densities. Fig. 3 shows that, as anodising proceeds at 24 A./sq. ft., there is some dissolution of the anodic film's outer surface in the electrolyte.

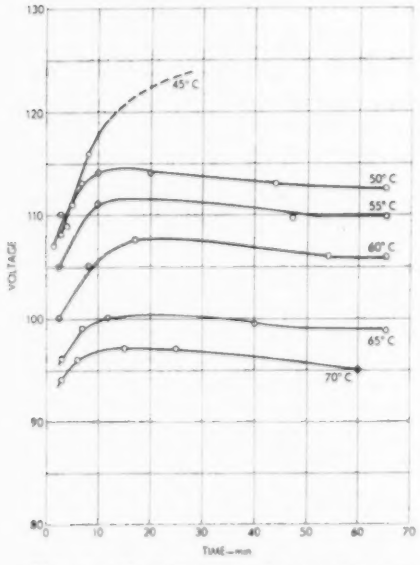

Fig. 3.—Variation of voltage with anodising time at various bath temperatures: malonic acid (12·5% w/v); current density 24 A./sq. ft.

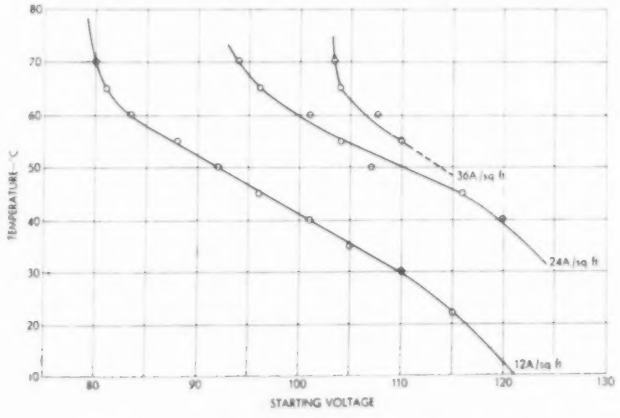

Fig. 4.—Variation of starting voltage with bath temperature at various current densities: malonic acid (12·5% w/v).

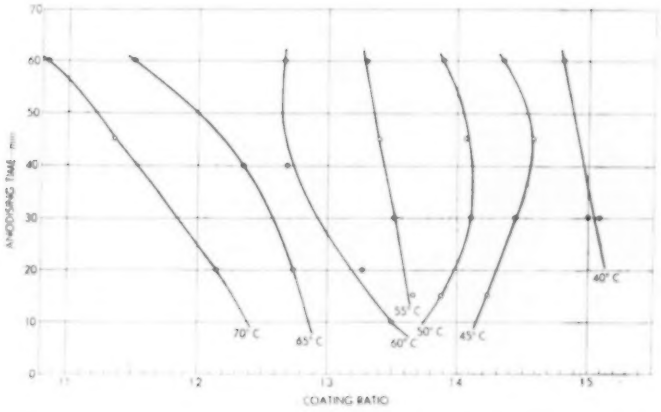

Fig. 5.—Variation of coating ratio with anodising time at various bath temperatures : malonic acid (12·5% w/v) ; current density 12 A./sq. ft.

as shown by the fall of voltage. Moreover, both Figs. 3 and 4 show that the over-all porosity of the anodic film will be much increased by an increase in temperature at any current density, owing to the attack by the electrolyte on the pore walls—the voltage rise during anodising becoming less as the temperature increases.

The value of the coating ratio[5, 6] obtained during an anodic process is of considerable importance in elucidating the optimum working conditions of the electrolyte employed, as far as current density, temperature, and time of anodising are concerned. The series of weighings carried out on the panels was as follows :—

wt. of panel before anodising .. .. *a*
wt. of panel after anodising .. .. *b*
wt. of panel after sealing .. .. *d*
wt. of panel after stripping .. .. *c*

$$\text{The coating ratio is therefore} = \frac{\text{wt. of oxide formed}}{\text{wt. of aluminium dissolved}} = \frac{b-c}{a-c}$$

The coating ratio is plotted against the anodising time for various current densities in Figs. 5, 6 and 7, and it is seen that at current densities up to and including 24 A./sq. ft., the coating ratio increases as the current density increases at any one temperature. As the temperature increases, the coating ratio decreases for all current densities, and the coating ratio also decreases as the anodising time increases : the tendency for this to occur is greater the higher the temperature. The only exceptions to this latter case appear to be the 12 A./sq. ft. coatings produced at 50 and 45° C., which show a maximum value of coating ratio after about 45 minutes anodising.

The results of these experiments are in agreement with both the appearance of the panels and the published work of Mason and Slunder.[6] These authors state that electrolytes used at elevated temperatures and with low current densities promote " light-weight " anodic films, and also that a long-time treatment in the electrolyte tends to decrease the efficiency of the oxide coating formation.

The appearance of the anodised material was of interest—a " light-weight coating " being formed when either the current density was low or the temperature high ; in particular when these two factors were conjointly operative. Such coatings were light yellow or yellowish-white in colour, and were formed as a result of the longer time that any one area of the coating had in contact with the anodising electrolyte. The longer this time was, and the hotter the electrolyte, the greater the solution of the film by the electrolyte, and the lower the coating ratio. The weight of the coating could be increased by increasing the current density—this causing the film to be formed rapidly and allowing less time for the film, at any one stage, to be in contact with the electrolyte. From this, it transpires that the highest coating ratios in malonic acid anodising will be formed using a cool electrolyte and a high current density for a short anodising time. Fig. 6 shows that, using a 12·5%

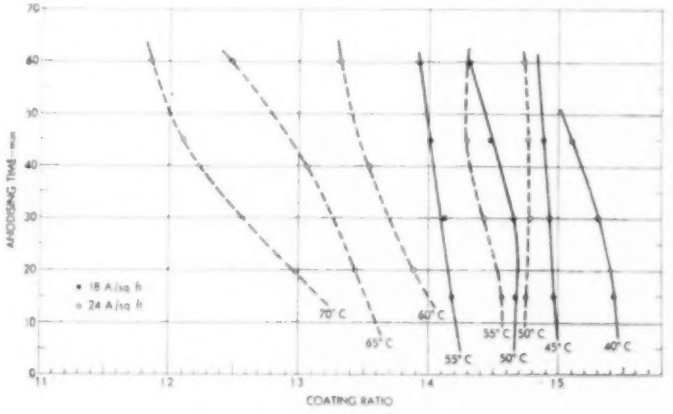

Fig. 6.—Variation of coating ratio with anodising time at various bath temperatures : malonic acid (12·5% w/v) ; current densities 18 A./sq. ft. and 24 A./sq. ft.

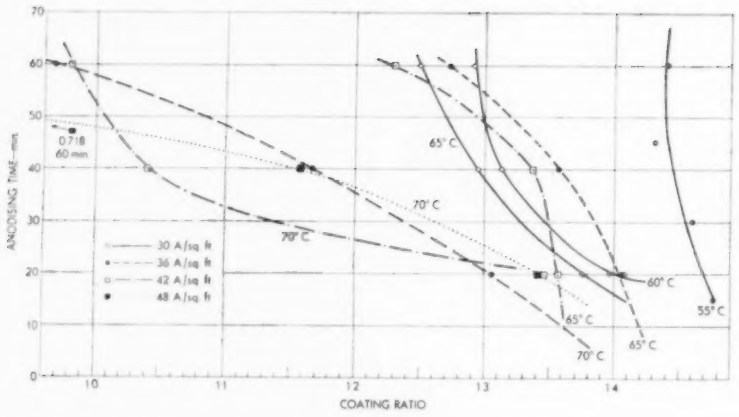

Fig. 7.—Variation of coating ratio with anodising time at various bath temperatures and various current densities : malonic acid (12·5% w/v).

w/v electrolyte, the coating ratio has a value of over 1·54, with a current density of 18 A./sq. ft. (the maximum permissible at a temperature of 40° C.). Only at 50° C. is 24 A./sq. ft. possible for any length of time, and it is seen that the increase in coating ratio made possible by the 6 A./sq. ft. increase in current, is more than compensated for by the 10° C. increase in electrolyte temperature, the coating ratio after 15 minutes' anodic treatment being barely 1·47. Similar comparisons may be made for the variables examined, using Figs. 5–7, and it appears that anywhere between 40° C. and 55° C. is suitable for the use of the 12·5% w/v electrolyte with current densities in the range 15–24 A./sq. ft. The uniform appearance of panels produced by such methods confirms this statement.

Fig. 8 shows a plot of the coating ratio against various operating temperatures for coatings produced in 60 minutes, using three different current densities. The graph clearly illustrates the superiority of the 24 A./sq. ft. coatings over those produced at 12 A./sq. ft. over the whole range of temperatures, but shows a current density of 30 A./sq. ft. to lower the coating ratio considerably, the higher the temperature, possibly due to a secondary anodic reaction (i.e. the rapid decomposition of malonate ion).

The increase in weight on sealing was expressed per 100 g. of anodic film and, if all the anodic film is converted to böhmite in accordance with the equation

$$Al_2O_3 \xrightarrow[\text{boiling water}]{\text{sealing in}} Al_2O_3.\,H_2O,$$

then 100 g. of anodic film increases in weight by 17·73 g. Fig. 9 shows the result of plotting the coating ratio against the percentage gain in weight on sealing. The curve indicates that, even at very high coating ratios, such as are obtained by using a cool electrolyte and a high current density, the percentage increase in sealing weight tends towards a minimum of 3%. In other words, the pore structure of such coatings is always such as to allow some penetration of water and hydrolysis to böhmite. Gains in weight on sealing greater than 6% were, in general, obtained with anodic films formed in 45 and 60 minute anodising times in the high temperature electrolyte. The 24% gain was a 60 minute, 48 A./sq. ft., 70° C. coating. This effect is due to attack by the hot electrolyte on the pore walls of the oxide, probably leading to pores of a funnel shape at the surface of the anodic film. This structure allows easy penetration of water molecules into the pores and subsequent hydration of a great proportion of the coating It is possible that, above 60° C., attack by the electrolyte is increased by some decomposition of the malonic acid to acetic acid, either by hydrolysis ($HOOC.CH_2.COOH \rightarrow CH_3.COOH + CO_2$) or cathodic decomposition.

Hardness and Abrasion Resistance

A series of commercially pure aluminium cylinders

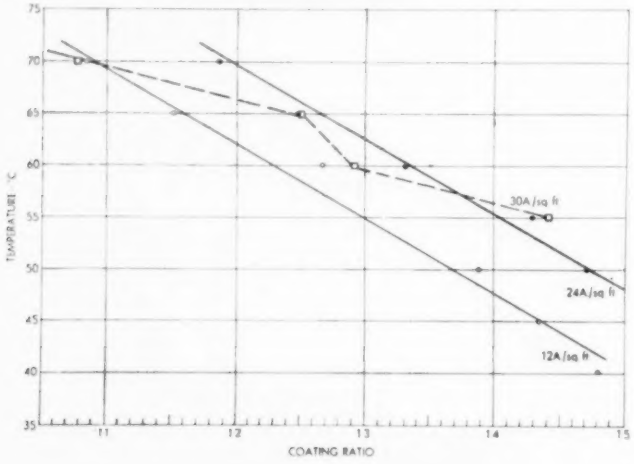

Fig. 8.—Variation of coating ratio with bath temperature at various current densities : malonic acid (12·5% w/v) ; anodising time 60 minutes.

TABLE I.—HARDNESS AND ABRASION RESISTANCE OF COMMERCIALLY PURE ALUMINIUM CYLINDERS ANODISED IN MALONIC ACID (12·5% w/v)

Temperature (°C.)	Time of Processing (min.)	Current Density (A./sq. ft.)	Coating Thickness (in.)	Carborundum to Penetrate Coating (g.)	Carborundum to Penetrate 0·001 in. of Coating (g.)	Loss of Weight of Coating (per g. of Carborundum)	Volume of Coating Removed per kg. of Carborundum (ml.)	Mean Hardness (V.P.N.)	Gain in Weight of Coating on Sealing (%)
55	60	12	0·001	274	274	0·0022	0·0030	459	2·87
55	45	12	0·0006	99	165	0·0015	0·0056	455	1·9
55	15	12	0·0001	36	360	0·0004	0·0041	—	—
55	30	12	0·0003	91	303	0·0009	0·0037	—	1·1
55	60	24	0·0015	385	256	0·0040	0·0038	414	2·56
55	15	24	0·0004	105	262	0·0009	0·0033	—	2·48
55	45	24	0·0011	278	253	0·0030	0·0040	443	2·29
55	30	24	0·0006	183	305	0·0014	0·0028	515	5·35
55	60	36	0·0024	794	322	0·0087	0·0049	367	5·66
55	15	36	0·00035	168	480	0·0009	0·0020	—	4·88
55	45	36	0·0015	511	329	0·0044	0·0032	401	2·67
55	15	36	0·0004	157	390	0·0010	0·0023	492	8·96
55	30	36	0·0010	332	315	0·0029	0·0032	402	1·33
45	60	12	0·0007	274	391	0·0016	0·0022	532	1·9
35	60	12	0·0007	259	370	0·0015	0·0021	432	1·9
65	60	12	0·0007	181	259	0·0018	0·0037	366	4·08
75	60	12	0·0006	100	154	0·0038	0·0147	—	4·72
70	60	12	0·0007	153	219	0·0027	0·0067	230	7·32
80	60	12	0·0004	44	110	0·0020	0·0168	—	16·41
Not Anodised		—	—	—	—	0·0028	0·0014	32	—

0·6 in. high and 1 in. in diameter were machined from forged bar and polished on one face to a mirror finish. They were then anodised at various times and temperatures as indicated in Table I, using the 12·5% w/v malonic acid electrolyte.

Table I also contains figures for the abrasion resistance of the coatings expressed in two different ways. The apparatus used was in accordance with the Schuh & Kern method described in B.S. 1615 : 1949, Appendix D. A standard coating prepared in accordance with the method described in Appendix C had an abrasion resistance of 22 g.

The cylindrical specimens, after the anodic treatments described in Table I, were sealed in boiling distilled water for 30 minutes and weighed, after drying and wiping off the surface bloom with cotton wool. The thickness of the anodic coating on each specimen was then carefully taken with an isometer. These figures are recorded in Column 4 of Table I, and are the average of three readings at three different places on the surface of the specimen, which had been previously polished. The cylinders were then abraded with 150 mesh Carborundum, exactly as described in Appendix D of B.S. 1615 : 1949, except that the air pressure was 15 cm. of mercury. When the coating was penetrated, the specimens were reweighed and the weight of Carborundum used to test each specimen, recorded (Column 5, Table I). Knowing the thickness of the coating, the specific abrasion resistance could be calculated, and is expressed as g. of Carborundum required to penetrate 0·001 in. of coating (Column 6). *Assuming* the density of a coating to be 2·5, and if the weight of Carborundum required to penetrate is known, then the abrasion resistance may be expressed as volume (in ml.) of coating worn away by 1,000 g. of abrasive (Column 8).

The Vickers hardness of the coating was determined prior to weighing the specimens on the first occasion. A Vickers projection microscope accessory was employed, using a load of 50 g. With some of the coatings it was extremely difficult to see the impression, so a 100 g. load was employed. With a few of the specimens the impression was invisible with any load. The loading time was 15 seconds and the results in Column 9 are the average of ten readings. A load of 20 g. was employed for the unanodised specimen.

Fig. 10 is a plot of surface hardness against the anodising time, and shows that the hardness diminishes considerably as the anodising time increases—allowing more attack by the electrolyte on outer layers of the anodic film as it grows, and thus promoting an increasing

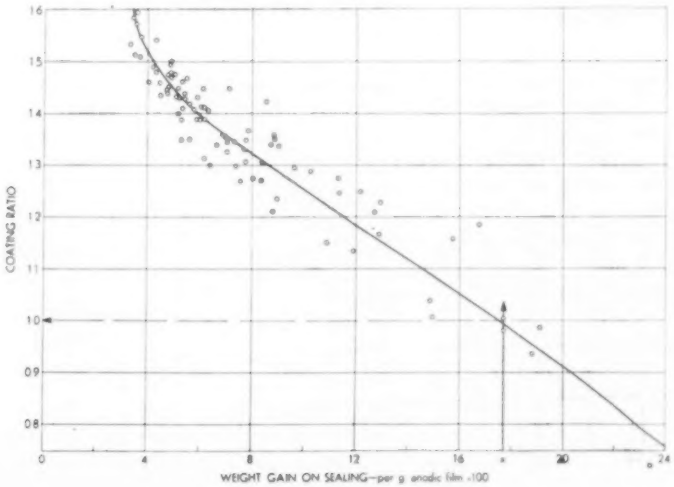

Fig. 9.—Variation of coating ratio with weight gain on sealing: malonic acid (12·5% w/v); various bath temperatures and current densities.

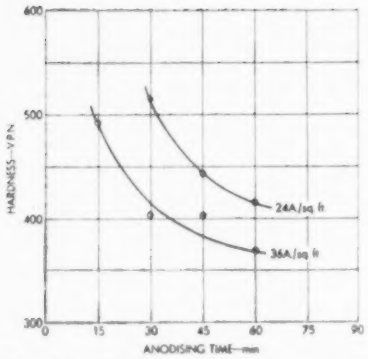

Fig. 10.—Variation of hardness with anodising time: malonic acid (12·5% w/v); bath temperature 55° C.

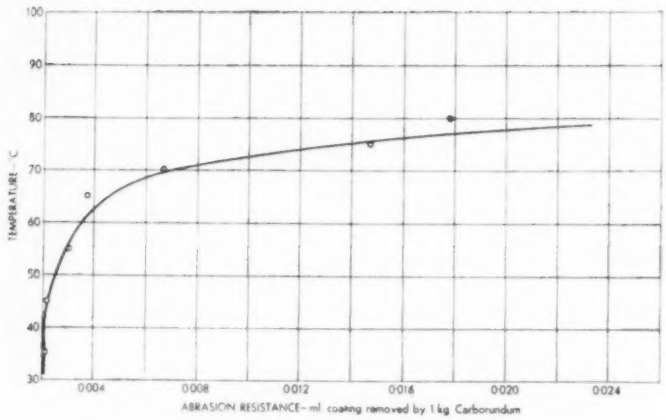

Fig. 11.—Variation of abrasion resistance with bath temperature: malonic acid (12·5% w/v); current density 12 A./sq. ft.

surface pore volume. The specific abrasion resistance figures showed the same tendency, but, as expected, the relationship was not clear cut, owing to the fact that the abrasion tests record an average figure for the whole coating, whereas the surface hardness figures are a measure of the condition of the outer layers. The hardness values of the 15, 20 and 45 minute coatings were very high—equivalent to those which had been found on hard-anodised surfaces.[8]

Figs. 11 and 12 record the variation of abrasion resistance with anodising temperature. A percentage increase in weight on sealing of up to about 4% causes little change in the abrasion resistance expressed in ml. coating worn away by 1 kg. of abrasive. However, at 65° C. and above, the abrasion resistance decreased at a very rapid rate with anodising temperature—as illustrated in Fig. 11. Fig. 12 shows the same effect—the specific abrasion resistance at 80° C. being approximately a quarter its value at 40° C. Unfortunately, not enough readings could be obtained to ascertain whether the same effect was operative with surface hardness, but it appears from Table I that the tendency is for this property to decrease appreciably with anodising temperature at 70° C. and above.

Use of the 12·5% w/v Electrolyte with Various Alloys

Alloys other than commercially pure aluminium may be processed in a 12·5% malonic acid electrolyte. A large variety of different material specifications was anodised, and it was noted that the coating colour and type of voltage behaviour were very similar to those when oxalic acid was employed. Examples of the behaviour of some alloys are given in Table II. and it

TABLE II.—BEHAVIOUR OF VARIOUS ALUMINIUM ALLOYS ON ANODISING WITH 12·5% MALONIC ACID

Alloy	Current Density (A./sq. ft.)	Time of Processing (min.)	Temp. of Processing (°C.)	Voltage Range	Film Thickness (in.)	Coating Colour
NT3 ½H ..	12	30	..5	115–120	0·0002	Opaque yellowish-white.
NS4 ½H ..	12	30	40	101–110	0·00025	Pale Yellow
NS4 ½H ..	24	60	50	105–110	0·0012	Brown
HE9 W ..	15	30	55	110	0·00015	Pale yellow
HE9 W ..	15	60	55	110	0·0004	Light brown
HE20 WP	15	30	55	120	0·0002	Opaque whitish-yellow
HS10 W ..	24	30	55	120	0·0002	Opaque whitish-yellow
H15 WP ..	12	30	65	68–120	0·00006	White
H15 WP ..	12	30	70	66–84	0·0001	White
D.T.D. 683	12	30	55	70–84	0·00025	Opaque whitish-yellow
D.T.D. 683	24	60	55	106–120	0·001	Opaque off-white
5½% Silicon Alloy ..	24	30	55	115–120	0·0002	Grey-yellow

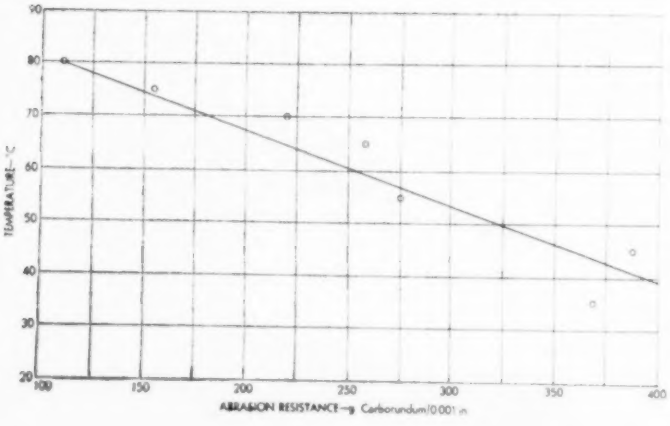

Fig. 12.—Variation of abrasion resistance with bath temperature: malonic acid (12·5% w/v); current density 12 A./sq.ft.

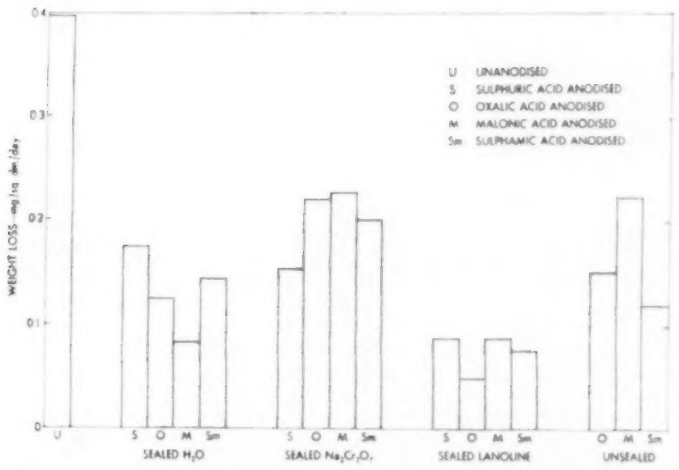

Fig. 13.—Variation of corrosion resistance with anodising and sealing methods.

should be noted that higher voltages are required to process commercially pure aluminium and alloys containing silicon, manganese and copper, and lower ones for magnesium and zinc. All alloys were suspended in the bath with commercially pure titanium wire.

Corrosion Resistance

Three sets of experiments were designed to test the resistance of the malonic acid anodised coating to corrosion. These were :—

(*i*) Comparison of H10 W material anodised and sealed by various methods. Resistance to 9 months' intermittent immersion in 5% salt water.

(*ii*) Comparison of HF15 WP and D.T.D. 683 alloys anodised various ways. Resistance to 4 months' intermittent immersion in salt water.

(*iii*) Galvanic corrosion tests on variously anodised NS4 ½H brass couples. Resistance to 5% salt water.

(*i*) *H10 W* (*Si* 0·97%, *Fe* 0·39%, *Mn* 0·53%, *Cu* 0·05%).

Sets of panels—5 in. × 2 in. × 10 s.w.g. were etched in 10% NaOH for ten minutes at 60° C. and then anodised as detailed in Table III. Sets of duplicate panels anodised by each of these methods were then sealed in the following solutions :—

(1) Boiling distilled water *p*H 5·6, 30 minutes (H).

(2) Boiling 5% w/v sodium dichromate solution, 15 minutes (C).

(3) Lanoline, 90° C. (panels heated to 90° C. before dipping), 1 minute (L).

The panels were removed from test after 9 months' intermittent immersion in salt water at room temperature and allowed to stand in cold concentrated nitric acid for 30 minutes to remove excess corrosion product. They were dried and weighed and the number of pits over 1 mm. diameter determined. Figs. 13 and 14 record the results of the experiments in the form of block graphs.

TABLE III.—ANODISING OF CORROSION TEST PANELS IN H10–W ALLOY

Anodising Bath	Time of Processing (min.)	Temperature of Processing (°C.)	Current Density (A./sq. ft.)	Code	Coating Thickness (in.)	Coating Ratio
Sulphuric Acid (10% w/v)	30	18	24	S	0·0005	1·522
Oxalic Acid (5% w/v)	30	18	24	O	0·0004	1·431
Malonic Acid (12·5% w/v)	30	55	24	M	0·0002	1·371
Sulphamic Acid (15% w/v)	30	40	15	Sm	0·0003	1·309

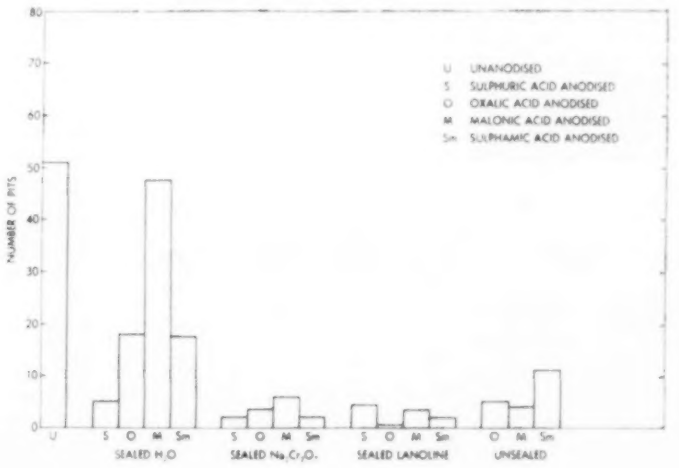

Fig. 14.—Variation of number of corrosion pits with anodising and sealing methods.

TABLE IV.—ANODISING OF CORROSION TEST PANELS IN HF15 WP AND D.T.D. 683 ALLOYS

Alloy	Anodising Bath	Time of Processing (min.)	Temperature of Processing (°C.)	Current Density (A./sq. ft.)	Coating Thickness (in.)	Coating Ratio
HF15 WP	Malonic Acid (12·5% w/v)	30	65	12	0·000864	0·522
	Chromic Acid (3% w/v)	according to D.T.D. 910 C*			0·00092	0·930
D.T.D. 685	Malonic Acid (12·5 w/v)	30	55	12	0·00925	1·120
	Chromic Acid (3% w/v)	according to D.T.D. 910 C*			0·00012	0·921

* Chromic acid anodising according to D.T.D. 910 C involves an increase in voltage from 0 to 40 in the first 10 minutes, 30 minutes at 40 V., an increase from 40 to 50 V. in 5 minutes, and 5 minutes at 50 V. The temperature is 40±1°C.

(*ii*) *HF15 WP and D.T.D. 683*

Sections of forged block were machined to panels approximately 2 in. × 3 in. × ¼ in. and anodised as detailed in Table IV. The panels were sealed for 30 minutes in boiling distilled water, weighed, and intermittently immersed for 4 months in 5% salt water at room temperature: unanodised test pieces were also exposed. After corrosion test, the panels were cleaned by immersion in cold concentrated nitric acid for 30 minutes, dried and weighed. In Table V is given the corrosion weight loss in mg. sq. dm. /day. On sectioning the panels, the grain structure was found to differ in some instances, so the results in Table V must be interpreted with caution, but are broadly illustrative of the protective value of the coatings.

It was noted, in the case of the malonic acid anodised materials, that all the corrosion losses were concentrated in highly local areas, with subsequent fairly deep pitting.

(*iii*) *Galvanic Corrosion Tests*

Sheets 2½ in. sq. NS4 ½H, 16 s.w.g., were drilled centrally with a ¼ in. diameter hole, and duplicate panels were anodised in the various electrolytes as indicated in Table VI, after etching for 3 minutes in 10% w/v caustic soda and dipping in concentrated nitric acid. The panels were sealed in boiling distilled water for 30 minutes, dried and weighed. Weighed 0 BA brass nuts and bolts were screwed into the central holes of each panel, finger-tight, and the panels were intermittently dipped in 5% salt water for a period of three months. After testing, the nuts and bolts were removed from the panels and the latter cleaned by dipping for 30 minutes in cold, concentrated nitric acid. The nuts and bolts were rinsed in water and freed of corrosion product by dipping for 2 minutes in cold 50–50 hydrochloric acid. Panels, nuts and bolts were weighed, after drying. The panels were then sectioned and the maximum depth of pitting, both in the region of the central hole and elsewhere, measured. The results are recorded in Table VII.

Once again the results should be interpreted with caution. There were two types of pitting attack in the region of the screw, probably determined by the stress applied when this was tightened. As a result, there was a considerable scatter in the pit depths. On the average however, it can be seen, once again, that the malonic coating stimulates deep local pitting, concentrating the corrosion loss into small areas.

TABLE VI.—ANODISING OF PANELS IN NS4 ½H ALLOY FOR GALVANIC CORROSION TESTS

Anodising Bath	Time of Processing (min.)	Temperature of Processing (°C.)	Current Density (A./sq. ft.)	Coating Thickness (in.)
(*a*) Malonic Acid (12·5% w/v)	30	40	12	0·0002–0·0003
(*b*) Malonic Acid (12·5% w/v	60	50	24	0·0012–0·0013
(*c*) Oxalic Acid (5% w/v)	10	20	15	0·0002
(*d*) Oxalic Acid (5% w/v)	60	20	15	0·001–0·0012
(*e*) Sulphuric Acid (10% w/v)	10	20	15	0·0002
(*f*) Sulphuric Acid (10% w/v)	60	20	15	0·001–0·0012

TABLE V.—CORROSION TESTS ON ANODISED HF15 WP AND D.T.D. 683 ALLOYS

Method of Anodising	Weight Loss by Corrosion (mg./sq. dm./day) (average of two readings)	
	HF15 WP	D.T.D. 683
Not Anodised	14·09	5·64
Malonic Acid (12·5% w/v)	3·99	0·17
Chromic Acid (3% w/v; to D.T.D. 910 C)	11·94	0·50

Dyeing and Pigmenting

A few trial experiments on pigmentation of standard anodic coatings revealed conclusively that the malonic acid coating produced at temperatures between 20–60° C. was very non-porous. For example, a series of panels, anodised to 0·0004–0·0005 in. in sulphuric, oxalic, malonic, chromic and sulphamic acid at 15 A./sq. ft., under normal anodising conditions, were pigmented with prussian blue and ferric oxide. The former process was carried out by double decomposition between 10% potassium ferrocyanide solution and 5% ferric nitrate at 25° C., the process being repeated once. The latter pigment was formed by immersion in a 20 g./litre solution of ammonium ferrioxalate at 40° C. for 5 minutes.

Deep blue and gold colours were produced in the sulphuric acid films, and pale shades in the chromic and oxalic coatings. No visible blue colour was produced in either the sulphamic or malonic coating, and only a very pale gold shade could be discerned. Similar effects were noted with dyestuffs, although, here, appreciable and visible quantities of dye were absorbed in the malonic acid coating. The amount of dye visible was dependent on the structure of the dyestuff, and the natural colour was masked by the intense yellow-brown colour of the coating.

A series of accelerated weathering tests was undertaken in order to compare the light fastness and weather resistance of dyed sulphuric, oxalic and malonic coatings. Samples of 16 s.w.g. NS4 ½H sheet were anodised as detailed in Table VIII, dyed in line with Table IX, and sealed for 30 minutes in boiling water. A strip of each panel was cut from the end and preserved as a standard in an envelope. Panels were exposed at 45° to an industrial atmosphere, facing S.W., and in a weatherometer test equipment, where they were subjected to 23 hours U.V. light from two 1 kW. sources and 22 hours distilled water spray each day. Periodically, specimens were removed from test and the fading rated according to the geometric grey scale—no fading = contrast 5, bad fading = contrast 1. Results for only a few of the dyes tested are given.

The results indicate that the oxalic and malonic acid dyed specimens are inferior to the sulphuric acid ones. This may be in proportion to the amount of dyestuffs absorbed into the coating and, if so, shows the decrease in porosity of the coatings in the order—sulphuric—oxalic—malonic. As the dyestuff fades, so the self-colour of the oxalic or malonic coating shows through, often causing the effect of darkening, but with

TABLE VII.—GALVANIC CORROSION TESTS ON PANELS IN NS4 ¼H ALLOY IN CONTACT WITH BRASS NUTS AND BOLTS

Anodising Bath	Coating Thickness (in.)	Forming Voltage	Average Loss in Weight of Panel (mg./sq. dm./day)	Average Gain in Weight of Nut and Bolt (mg./sq. dm./day)	Average Maximum Depth of Pitting Near Hole (in.)	Average Maximum Depth of Pitting Elsewhere (in.)
Not Anodised	—	—	104·6	8·9	0·0053	0·0012
Sulphuric Acid	0·0002	18	107·2	3·0	0·014	0·0006
Sulphuric Acid	0·0012	18	87·1	11·0	0·012	0·0018
Oxalic Acid	0·0002	60	97·1	3·9	0·014	0·001
Oxalic Acid	0·0012	60	91·3	2·8	0·019	0·00095
Malonic Acid	0·0002	102	92·9	5·4	0·017	0·0006
Malonic Acid	0·0012	105	92·1	1·3	0·019	0·00095

loss of depth of colour. The light fastness of the self-coloured anodic coatings is excellent.

DISCUSSION

The results of the preceding work indicate that a considerable variety of anodic films, having different physical properties, may be produced in an electrolyte containing 12·5% w/v malonic acid. Broadly speaking the malonic acid process appears to produce a film which theoretically consists of a thick barrier layer upon which rests a relatively non-porous outer oxide layer. The corrosion tests and absorption of dyestuffs described in this paper support a structure corresponding to this description, as given by the results in comparison with sulphuric, oxalic, sulphamic and chromic acid anodic films.

If operated at a low temperature and a high current density, the malonic acid process is capable of producing anodic films of a high surface hardness on copper-free alloys. As has been previously reported,[7] the film is considerably softer if the alloy contains zinc or copper. As the operating temperature is increased, the current density which it is able to pass increases, but the coating ratio decreases appreciably. It was found that the temperature was of considerably more importance than the current density in effecting a change of coating ratio. High operating temperatures promoted low coating ratios, and although this could be partially counteracted by passing more current density, there was a definite limit in the effectiveness of this countermeasure at any one temperature. Too high a current could decrease the coating ratio, possibly by promoting side reactions, such as the anodic oxidation of malonate ion.

As the temperature of the electrolyte was raised at any one current density, so the forming voltage and the rise in voltage on anodising diminished. However, even at 12 A./sq. ft., the forming voltage was still 80 at 70° C., and thus a thicker barrier layer was being formed than with an oxalic acid electrolyte at 20° C. The porosity of the outer layer of anodic film increased considerably as the anodising temperature increased, and this was shown by a plot of coating ratio against the gain in weight on sealing in boiling distilled water. At anodising temperatures below 55° C., the percentage gain in weight on sealing was rarely more than 6% at any current density. Thereafter, as the temperature increased, so did the percentage gain in weight on sealing, particularly for the thicker coatings produced by 40–60 minutes anodising. There also seemed to be a tendency for coatings anodised at 12 A./sq. ft. for 20 minutes to show a greater percentage increase in weight on sealing than those produced at the higher current densities in 20 minutes, although the position was reversed for 40–60 minutes anodising time.

These facts show that, as the temperature of the malonic acid electrolyte increases, so does the average

TABLE VIII.—ANODISING OF SAMPLES OF 16 S.W.G. SHEET IN NS4 ¼H FOR WEATHERING TESTS

Code	Anodising Bath	Time of Processing (min.)	Temperature of Processing (° C.)	Current Density (A./sq. ft.)	Operating Voltage	Coating Thickness (in.)
M	Malonic acid (12·5% w/v)	30	55	15	87–93	0·0005
O	Oxalic acid (5% w/v)	60	22	18	56–68	0·0012
S	Sulphuric acid (6·8% w/v)	60	20	15	18–20	0·0012

TABLE IX.—WEATHERING TESTS ON ANODISED AND DYED SAMPLES OF NS4 ¼H ALLOY

Details of Dyeing	Anodising Method Code*	Hours in Weatherometer				Months Out of Doors		
		100	500	1500	4000	3	6	8
15 Minutes in 5 g./l. Solution of G2LW Yellow at 60° C.	S	5	5	5	4	5	—	4–3
	O	5	5	4–3	3	—	5	—
	M	5	5	5–4	3	—	4	—
15 Minutes in 5 g./l. Solution of B3LW Red at 60° C.	S	5	5–4 Redder	4 Redder	4 Redder	5–4 Darker	—	4–3
	O	5	4	4	3	—	5–4	—
	M	5	4	3	2–1	—	4	—
15 Minutes in 10 g./l. Solution of MLW Black at 60° C.	S	5	5	5–4 Darker	4 Darker	5	—	4 Darker
	O	5	4	3	3	—	5–4	—
	M	5	4	3	3	—	3	—
15 Minutes in 8 g./l. Solution of GLW Green at 50° C.	S	5–4	4	4–3	4–3	—	—	—
	O	5	5	5	4–3 Darker	—	5–4 Darker Blue	—
	M	5	5	3 Bluer	3 Bluer	—	5–4 Darker Blue	—
15 Minutes in 0·5 g./l. Solution of LLWS Blue at 60° C.	S	5	5	5	5	—	—	5
	O	5	5	5	5	—	—	5
	M	5	5	5–4	5–4	—	5	—

* See Table VIII.

porosity of the outer oxide layer, but this may be partially offset by increasing the current density up to a given limit—depending on the temperature.

The thick-barrier-layer non-porous-outer-layer type of structure is obviously a system where, in a corrosive environment, relative to a given sealing treatment, very deep localised pitting may be promoted, owing to the very few sites at which corrosion can occur, since the corrosive ions must seep through the few channels in the outer oxide skin and finally penetrate a very thick non-porous barrier layer before they can attack the underlying metal. From a corrosion point of view, therefore, a coating formed in malonic acid may be found to be corrosion resistant on the assessment of attack by weight loss, but fall down because the weight loss is concentrated in a few local areas.

From such considerations it appears that the ideal possible protection of metal from corrosion, from the anodic film aspect, is given by a coating possessing a certain barrier layer thickness and a certain thickness of porous outer oxide, such that any corrosion is not localised, but, on the other hand, not such that a great weight of metal is lost on corrosion. Obviously, the formation of such an ideal coating is dependent on the alloy being processed, but on H10 W alloy the sulphamic acid anodic film seemed to fit the bill, with an intermediary forming voltage of around 40 and, assumedly, more or less, the correct film thickness of 0·0003 in. 0·0005 in. sulphuric acid anodising afforded 50% protection, as assessed by weight loss, and about 66% protection against pitting. 0·0002 in. malonic acid anodised coating was the other extreme—offering virtually no protection according to pitting attack assessments, and more than 75% on weight loss tests.

The possible structure of the malonic acid coating may be interpolated from the work of Keller, Hunter and Robinson,[8] which shows that the forming voltage determines the size and number of pores. Under normal anodising conditions, the forming voltages in sulphuric, oxalic and chromic acids are 20, 60 and 40 on 99·5% aluminium. The number of pores/sq. in. $\times 10^9$ are 334, 37 and 52, respectively, and the percentage pore volumes are about 6%, 2% and 4%, respectively. The wall thickness and barrier layer thicknesses are of the same order, being very approximately 160 Å, 600 Å and 440 Å, respectively. This produces models for the three coatings, approximately as illustrated in Fig. 15.

The models at least illustrate that, as regards corrosion resistance, the malonic acid coating will perform very well when the resistance is expressed as a weight loss, because of the few pores present through which the corrosive medium can penetrate, and the thick barrier layer. Obviously, breakdown of such a coating will occur very locally, where weak spots in the barrier layer coincide with pore bases, and such breakdown appears to lead to very concentrated localised attack. Owing to the lack of porosity, sealing in dichromate solutions is of little value, as any dichromate absorbed may easily be leached out owing to the large pore diameter. In the case of the sulphuric acid coatings, the dichromate is more easily absorbed and held in the large number of small diameter pores, and can exercise the effect of a corrosion inhibitor satisfactorily. It was noted at the conclusion of the salt dip tests that the salt solution was tinted yellow after dichromate-sealed malonic, oxalic and sulphamic coatings had been tested. Sealing in organic

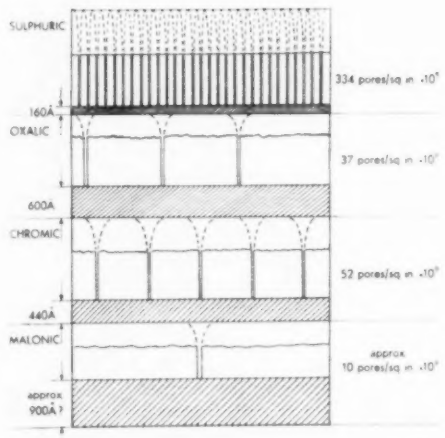

Fig. 15.—Diagrammatic sketches of coatings produced by anodising in sulphuric, oxalic, chromic and malonic acids.

media, such as lanoline, produces a coating fairly impervious to saline corrodants and obviously the less pores to fill with lanoline, the less chance of breakdown. Moreover, it will be easier to fill a large diameter pore, such as is present with oxalic or malonic acid coatings, than the small pores of the sulphuric coating.

Fig. 15 is also of interest in connection with the absorption of dyestuffs and pigments and the gain in weight on sealing in boiling water, all of which are determined by the size and number of pores at the outer layer of the coating. The shape of the pores at the surface is determined by the degree of chemical attack of the electrolyte on the pore walls during the formation of the anodic film. It is reasonable to suppose that the greater the solvent power of the electrolyte at any given temperature, and the greater the pore diameter, the greater the porosity at the surface—depending on the number of pores. At optimum temperatures of anodising—i.e. between 40 and 60° C.—the outer porosity of the malonic coating is obviously very small, as revealed by absorbtion of dyes and pigments and by the sealing gains recorded in Fig. 9, relative to the various coating ratios. As the temperature increases beyond 60° C., however, so the attack on the surface layers increases rapidly, and this fact is reflected by the rapid deterioration in abrasion resistance and surface hardness (Figs. 11 and 12). This may be due to decomposition of the electrolyte to acetic acid.

Coloration of malonic acid coatings by dyestuffs yields palely coloured surfaces tinted by the sepia brown or golden brown colour of the coating. The pale colours are presumably due to the few large pores, which disperse the dyestuff molecules away from each other across the surface. Naturally, little dyestuff is absorbed and the light fastness suffers.

CONCLUSION

The use of malonic acid as an anodising electrolyte has been established. It has been shown that the coating is very non-porous and can be produced in electrolytes of various concentrations, that of 12·5% w/v being favoured for this investigation. The application of the results of American work on the structure of the coating given by the malonic acid process indicates that this technique of anodising should be capable of producing a large range

of surfaces suitable for various applications. Extremely hard coatings, comparable to hard anodic films, may be obtained on commercially pure materials.

An investigation of the value of the coating ratio at various anodising temperatures and current densities shows that the latter decreases as the temperature increases and the effect cannot be wholly compensated for by raising the current density. The gain in weight on sealing in boiling water was equivalent (theoretical conversion) to böhmite when the coating ratio was about 1·000, which may explain why chromic acid coatings may be completely converted to böhmite,[9] as the coating ratio of such anodised films is around 1·0.

Self-coloured coatings obtained by anodising in malonic acid are very light-fast, but do not dye to produce permanent colours.

The corrosion resistance, as assessed by weight loss, obtained on malonic acid anodised aluminium is high, but there seems to be a tendency for intense local pitting to occur. On the other hand, coatings produced in sulphuric acid suffered shallow pittings with a high weight loss and it may be that a combination of a suitable barrier layer, porous outer layer and sealing would produce a coating of optimum corrosion resistant properties. The nearest approach to this was a coating produced in sulphamic acid and sealed in lanoline.

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Improving the Quality of Chromium Plating

IT is hoped that a new British Standard published recently will provide the starting point in a general upgrading of electroplated chromium finishes. The standard is called "Electroplated Coatings of Nickel and Chromium" and carries the number B.S. 1224 : 1959. Coinciding with the issue of B.S. 1224 comes news that a nation-wide labelling scheme is being launched so that buyers of chromium-plated products will be able to tell whether the plating is up to standard. A third development is the introduction of a new measuring instrument which will make it simpler for platers and manufacturers to check that their products comply with the standard.

New Standard

The new British Standard is a revision of an earlier specification, which has been revised, at the request of industry, as a step towards improvement in quality of chromium plating and an answer to the all-too-numerous complaints about the lack of durability of many plated surfaces. The most important change in the new edition of B.S. 1224 is a provision for thicker coatings of nickel. With most plated products it is the layer of nickel—rather than the top "flash" of chromium—which largely decides the quality and durability of the finish known as chromium plating. Until recently, nickel has been in short supply, but with the end of this shortage there is no longer any reason why an adequate nickel coating should not be applied.

A second important change is the inclusion of new, more stringent laboratory tests which plated products must be able to pass if compliance with the standard is claimed for them. Two of these tests are specially concerned to ensure that chromium plating will stand up to corrosion—a vital factor with, for example, bumpers and other automobile parts which are exposed to the weather. In all, nine tests are prescribed in the standard. They include the tests for corrosion resistance—referred to above—in which plating is exposed to the attack of sulphur dioxide, or acetic acid and salt; tests to check the thickness of both the nickel and chromium coatings deposited on an article; and tests to ensure that the plated finish will not easily peel away.

Platers and manufacturers will be glad to learn that in gauging the thickness of the layer of nickel which is called for in the standard, they may now use a non-destructive testing instrument developed by scientists at the British Non-Ferrous Metals Research Association. Hitherto, this assessment could only be made by testing a sample to destruction—an expensive process. The standard still quotes such methods, but also draws attention to the value of the new instrument for day-to-day control purposes.

Labelling Scheme

It is not possible for the man-in-the-street, just by looking, to differentiate between good and bad chromium plated finishes, but identification of products complying with the standard will be made possible thanks to a labelling scheme which, in the first instance, is being operated by the Mond Nickel Co., Ltd. Mond Nickel—working in close collaboration with the British Standards Institution—will supply labels to platers and manufacturers of plated products who must undertake to apply these labels only to plating which complies with B.S. 1224.

The new British Standard calls for three grades of plating, depending on the use to which plated products are put, and the Mond labels take this into account. They will be coloured red (which indicates suitability for "severe" service), blue ("normal" service), or green ("mild" service). For example, plating which is to be exposed for long periods in the open should have a red label; that used indoors but where corrosion is likely (in steamy kitchens and bathrooms, for example, or on products stored in damp cupboards) should bear a blue label. Where less arduous conditions are involved, the "green-for-mild" category will be quite sufficient. Each of the labels will announce that the nickel-chromium plate conforms to British Standard 1224 : 1959.

At this stage, it will be left to platers and manufacturers to satisfy themselves that their finishes are in accordance with the standard. Large-scale buyers (such as motor-car firms) will doubtless require samples to be tested before accepting delivery of goods.

Copies of B.S. 1224 may be obtained from the British Standards Institution, Sales Branch, 2, Park Street, London W.1. Price 5s.

Vertical Cylindrical Closed Quench Furnace

New Equipment for Gas Carburising and Carbonitriding

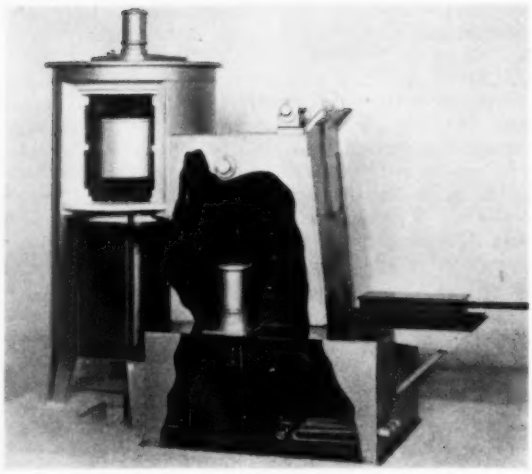
A NEW atmosphere furnace combining the advantages of pit type gas carburisers with facilities for closed quenching, hitherto available only in horizontal equipments, is announced by the Electric Resistance Furnace Co., Ltd. The new furnace does not supersede the Efco-Lindberg horizontal furnaces, but for some types of charges, particularly dense loads of parts having internal bores, and long components, it offers distinct advantages.

The furnace has a vertical cylindrical heating chamber which is closed at the top and has provision for the insertion of a charge through the base. The charge is shielded from direct radiation by a heat-resisting baffle suspended from the furnace roof or, alternatively, the sides of the charge basket. A powerful fan of large diameter, mounted directly above the charge, draws the atmosphere up through the charge and forces it down over the heating elements, ensuring uniformity of heating and intimate contact of the atmosphere with all surfaces of the charge.

Because of its cylindrical shape, the chamber accommodates a larger volume of work within a smaller wall area and operates at greater efficiency than an equivalent horizontal rectangular furnace, and an economy in gas or electricity consumption of the order of 15% is claimed. Atmosphere consumption is also reduced due to the smaller volume of heating chamber to be purged. The mouth of the heating chamber and bung are designed to form a heat trap.

The work, normally in a nickel-chrome basket, is moved into the furnace through an outer, vertical rising door to enter the purging vestibule. Here it stands on one of the two platforms of the quench elevator. At the outer door a gas curtain automatically prevents ingress of air when the door is open. Both the door and the quench elevator are pneumatically operated. After purging, the charge is transferred to the furnace bung where it is supported on suitable stools which allow free circulation of the atmosphere.

A feature of the furnace is the use of a charge trolley to move the charge horizontally from the outer loading table into the purging vestibule and to and from the furnace bung. The trolley runs on rail tracks and is fitted with wheels which are mounted on ball bearings and carried in an articulated frame to ensure easy movement. Built into the trolley is a raising and lowering mechanism operated from outside the furnace with a removable push rod. The trolley can be moved in and out of the furnace without raising the outer door, a swinging counter-balanced flap attached to the outer door being provided for this purpose. After charging, the trolley is lowered and withdrawn. The use of the trolley obviates the need for hearth rollers and, apart from the fan, no mechanical parts are subjected to furnace temperatures.



Cut-away model showing the work container in the cylindrical heating chamber on the left. On the right is the purge chamber with the quench tank below ; the charge trolley can be seen on the extreme right, outside the purge chamber.

Stops are provided to ensure accurate location of the charge on the quench elevator and on the furnace bung which is raised hydraulically to lift and seal the charge into the heating chamber. The charge is removed from the chamber by lowering the bung and transferring to the quench elevator by means of the charge trolley. When it has been lowered into the integral quench tank, the outer furnace door can be raised and a new charge inserted.

The purging vestibule and the space beneath the heating chamber are water cooled. The quench tank of adequate capacity is fitted with oil preheaters, a multi-tube water cooling system, and a two-speed reversible agitator and baffle arrangement to direct a large volume of oil over the charge being quenched. The agitator, its driving motor, and the cooling system are mounted on a removable top plate for easy access for maintenance. The furnace can also be supplied with a hot oil quench tank for marquenching, which is of larger capacity and fitted with special air cooled tubes.

When electrically heated the furnace chamber is fitted with patented Corrtherm elements, which are sheets of corrugated nickel-chrome covering almost the entire wall. The life of the elements is not affected by soot deposition from hydrocarbon gas because the voltage and watts dissipation are so low that leakage across the deposit is negligible. When gas fired, specially designed radiant tubes are positioned around the walls of the heating chamber. For easy maintenance the roof of the furnace chamber is removable and gives access to the fan and the elements or radiant tubes. Atmospheres for the carburising and carbonitriding processes are obtained from an EFCO endothermic generator operating on town's gas or liquid petroleum gas.

Low Pressure Metallurgy

A General Survey

By A. S. Darling, B.Sc. (Eng.), Ph.D., A.M.I.Mech.E.

The carrying out of metallurgical operations in vacuum has more to it than the prevention of oxidation. A metal or alloy concerned in a melting, heat treatment or refining operation may be considered as one component in a polyphase system. Pressure variations add a further dimension to the degrees of freedom previously represented only by temperature and concentration, with the result that equilibrium conditions can frequently be displaced in a favourable direction. In the first part of this general survey of low pressure metallurgy, published in October, the author discussed historical aspects. In the present instalment he deals with cathodic sputtering and vacuum evaporation, and the survey will be concluded next month with a consideration of vacuum melting.

(*Continued from page* 143 *of the October issue*).

CATHODIC SPUTTERING

THE glow discharge which develops when a high voltage is applied between electrodes in a partially evacuated system causes progressive disintegration of the cathode. The metal emitted leaves the cathode at high speed and the vapour cloud thus formed condenses on the nearest cold surface to produce a more or less adherent film. Cathodic disintegration must have been one of the first low pressure metallurgical processes to have been investigated in any detail. Even so, the mechanism involved is still not fully understood. Groves first observed the phenomenon in 1852.[45] Six years later, Pluecker[46] studied the effect on a more quantitative basis and confirmed that the condensed metal film accounted for the weight lost by the cathode. The present term, "sputtering", is derived from Pluecker's description of the effect as cathodic pulverisation (Zerstäubung).

Sir William Crookes pioneer work on the passage of electricity through gases included a comprehensive experimental study of cathodic pulverisation.[47] His experiments were carried out in a reduced pressure of air, and under these conditions it was shown that platinum and the other noble metals sputtered very rapidly compared with aluminium or magnesium. These results were confirmed by the later researches of Kohlschütter,[48,49] who used nitrogen as a working gas and showed that silver, gold, platinum, palladium, copper and nickel had sputtering rates in descending order of magnitude. Although the process had a rather limited application at this period, it was of great theoretical interest and attracted considerable attention from physicists. Much of this early work was reviewed by Kohlschütter in 1912.[50]

It was appreciated at an early date that sputtering could be carried out under a wide range of conditions. The pressures involved could vary from 5 to 1,000 microns, and working voltages ranged from 500 to 20,000. General agreement prevailed that with the same cathode and the same gas the rate of disintegration was proportional to the current and increased with the cathode fall. So long as the cathode fall was "abnormal", the pressure of gas in the system was of importance only in so far as it affected these two factors. The effects caused by traces of extraneous gas in the system were also recognised. It was known, for example, that whereas sputtering proceeded particularly rapidly in pure argon, and at a somewhat lower rate in neon, the presence in either of these noble gases of <1% of a more reactive impurity reduced considerably the rate of disintegration. Mercury vapour, in spite of its high molecular weight, was particularly effective in this respect.[51]

A fairly detailed study by the staff of the G.E.C. laboratories at Wembley was intended to elucidate the basic mechanism of sputtering: the results of this work were published in 1923.[52] An uncertain factor in previous investigations had been the velocity with which the positive ions struck the cathode. Ions were normally produced everywhere in the vessel, and it was realised that the speed at which they approached the target depended upon the difference of potential between the cathode and the region in which they were formed.

The G.E.C. workers minimised this uncertainty by studying the discharge effects in a triode with a thermionic cathode. The target was in the form of a tungsten spiral which was used as a grid and maintained at a potential negative with respect to that of the heated cathode. The rate of sputtering was simply determined by measuring the rate of increase of resistance of the spiral. This operation could, of course, be carried out without breaking the vacuum or disturbing the experiment in any way. The current collected by the grid was a direct function of the number of ions falling upon it. When the mean free path of the gaseous ions was greater than the dimensions of the vessel, the ion velocity could be directly related to the nature of the gas and conditions of discharge in the system.

It was found that the number of atoms of tungsten discharged by each impinging positive ion was independent of the gas pressure, providing this was below about thirty microns. At higher pressures the proportional rate of disintegration decreased as the pressure increased. The rate of disintegration was independent of the temperature of the grid and increased in a linear fashion with the applied voltage. The sputtering rate varied with the nature of the gas employed and increased in the order: hydrogen, helium, nitrogen, neon, mercury and argon. No definite theory of disintegration was proposed in this paper. It was concluded, however, that the primary mechanism of disintegration occurred at energy levels not greatly exceeding those required to initiate ionisation of the gas.

Primary and Secondary Sputtering

On the basis of his extensive researches on this subject, Güntherschulze has distinguished between the mecha-

nisms of "primary", and "secondary" sputtering. Primary sputtering is that which occurs at low pressures and low currents. It corresponds to the disintegration occurring in the low pressure range discovered by the G.E.C. workers, in which the specific sputtering rate was unaffected by gas pressure. Since light currents only can be employed under low pressure conditions the total rate of disintegration is low. "Secondary" sputtering is therefore the mechanism of technical importance. It is usually carried out at pressures of several hundred microns and heavy currents are employed. In spite of the fact that the total rate of disintegration is thereby greatly increased, the process is far less efficient than that of primary sputtering.

Information concerning the basic mechanism of cathodic disintegration is obviously more easily attainable if the experiments are carried out under conditions favouring the primary process. The gas pressure is then kept so low that it can be neglected. Secondary sputtering is usually carried out under glow discharge conditions, and the gas pressure is then a very important factor. It is responsible for determining not only the velocity with which the ions strike the cathode surface, but also the subsequent movements of the metal atoms discharged.

Güntherschulze's early work[53] soon indicated that the G.E.C. investigators had arrived at conclusions which were faulty in two respects. Firstly, they had been in error about the rate of disintegration in helium. Güntherschulze's experiments, the results of which have since been generally confirmed, showed that if the helium were pure no sputtering occurred: the G.E.C. workers must therefore have used impure gas. The second point concerned the shape of the curves relating the rate of sputtering to the applied voltage. In this low pressure range, when specific rates were independent of gas pressure, the G.E.C. workers had found a linear relationship between voltage and rate of disintegration. Güntherschulze showed that the primary sputtering rate was a square-root function of the applied voltage.[54] Secondary sputtering rates, however, were found to vary in a linear manner with the cathode fall.[55]

Two theories explaining the mechanism which permitted positive ion bombardment to disintegrate solid metal were current at this period. One school of thought maintained that the effect was simply one of thermal evaporation from regions of high local temperature. Difficulties were experienced in reconciling this hypothesis with the experimental fact that the rate of disintegration did not vary appreciably with the cathode temperature.[52] The second theory was one which envisaged a mechanical "bumping-off" of surface atoms by ions which penetrated beneath the cathode surface and then rebounded to transfer a part of their outward momentum to metal atoms which they struck from behind. Langmuir had shown that the latter mechanism was consistent with the threshhold kinetic energy which the ions required to initiate sputtering. Lamor and Compton[56] proposed a special theory of cathode sputtering in 1934 which involved the combined operation of these two mechanisms. Light ions, such as those of hydrogen were known to penetrate the cathode more than larger heavier ions. While the two mechanisms were supposed to be complementary, the bumping-off process was expected to be the predominant one when metal was being sputtered by small ions, whereas the evaporation process was likely to be of greater importance when the ions were large. The experimental evidence available was, in general, not sufficiently detailed to conform the accuracy of this modified theory.

In a recent paper, Güntherschulze[57] indicated how many of his earlier experimental results could be reinterpreted in a manner which reconciled several apparent anomalies. The different shapes of the curves connecting sputtering rates with applied voltage had led to a belief that primary and secondary sputtering were caused by distinct mechanisms. Primary sputtering varied as the square-root of the applied voltage. It could therefore be concluded that the rate of sputtering depended upon either the velocity or the momentum of the bombarding ion. Secondary sputtering, on the other hand varied directly as the cathode drop. This had been generally accepted as evidence that the sputtering rate depended upon the kinetic energy of the impinging ion.

Güntherschulze emphasised that this conclusion rested on the faulty assumption that the kinetic energy of the positive ions was proportional to the cathode fall. In actual fact, however, not only did the ion energy increase at a greater rate than the cathode fall, but the greater velocity with which the metal atoms were emitted at the higher voltages caused a movement of the neutral zone away from the cathode surface. These two factors working in conjunction meant that the kinetic energy of the bombarding positive ions could not be derived from the cathode fall alone.

Secondary sputtering rates, therefore, when plotted against the true striking energy rather than the cathode fall, gave curves which had a square-root dependence virtually the same as that observed in primary sputtering. This agreement was further enhanced when the sputtering rates were corrected to make allowance for the presence of an electron current. Güntherschulze showed in 1930[58] that the threshhold energy for electron emission was identical with that required to initiate sputtering.

It appears reasonable to assume, therefore, that both primary and secondary sputtering depend upon the same basic mechanism, which ensures that the specific sputtering rate varies with the velocity or momentum of the impinging ions rather than their kinetic energy. The essential difference between the two processes is one of efficiency, since it has been conclusively demonstrated that the atoms emitted per unit striking ion decrease when the pressure exceeds a critical value. The conditions under which secondary sputtering proceeds are remarkably complex. The positive ions are subjected to continual collisions with gas molecules during their passage towards the cathode, so that they strike their target with a somewhat uncertain velocity. This leads to further uncertainty concerning the velocity of the emitted metal atoms. These atoms lose their original direction because of gas molecule collisions soon after leaving the cathode surface, and tend to form a neutral cloud of metal vapour which retards still further the motion of the impinging ions. Some atoms from the vapour cloud diffuse to the nearest cold surface and condense, but many migrate back to the cathode surface. A considerable deposit of metallic dust is, in fact, frequently found on cathode surfaces at the conclusion of an experiment.

Methfessel[59] claims that, by using a cathode in the form of a wire mesh, back diffusion effects can be minimised and sputtering rates considerably increased.

In one instance quoted, a cathode consisting of meshed silver wire facilitated the deposition, in two minutes, of a film which normally required three hours with a flat plate cathode. This tremendous increase in sputtering rate appears to be exceptional, however, because the somewhat limited experimental evidence available suggests that specific sputtering rates under "primary" conditions are only about one order of magnitude higher than those under "secondary" conditions. Some diffusion calculations of Güntherschulze,[57] which indicate that primary sputtering should theoretically be approximately one hundred times faster than secondary sputtering, do not reconcile the anomalies.

Problems of efficiency are involved, however, even when the mechanism of primary sputtering is under consideration. Recent work by Keywell[60] has demonstrated that with an applied potential of 3,480 volts, 7·9 silver atoms were sputtered for each incident positive ion. Argon ions accelerated through this potential, however, have an energy sufficient to account for the emission of 975 silver atoms. The author concludes that the argon ions lose their energy in the silver target by a diffusion-collision process, and that the results obtained could be interpreted in terms of the neutron diffusion theory. Keywells' specific sputtering rate values are comparable in magnitude to those of Meyer and Güntherschulze[54] and Penning and Moubis.[61]

Agreement between the results of various sputtering experiments carried out under low pressure conditions is, in fact, reasonably satisfactory, and information dealing with sputtering rates under industrial conditions is, although somewhat scanty, fairly concordant. Data pertaining to experiments carried out with the same gas and the same metal over a wide range of pressures, which would permit some sort of correlation between the primary and secondary processes, is not available however.

Laboratory Applications of Sputtering

Sputtering was found at an early date to provide a convenient method for the production of conducting films on glass, silica and other non-metallic materials. Whereas silver was the only metal which could be conveniently deposited by chemical methods, sputtering permitted the ready deposition of all metals except magnesium and aluminium upon a non-conducting surface. The method was used in the early years of the century for silvering and platinising the quartz fibres of electroscopes and string galvanometers, and the necessary techniques were well established by 1925 when Mazur[62] published the results of his experiments on the microstructure of metallic surfaces which had been subjected to cathodic bombardment. He was able to show that the structures produced by this technique were the same as those obtained by chemical etching methods. The material sputtered from two-phase alloys such as those of cadmium and bismuth had a composition different from that of the parent surface.

C. S. Smith,[63] unaware at this time of Mazur's work, decided in 1925 to investigate the possibilities of cathodic etching as a metallographic technique. He found that the process, which delineated in a very satisfactory manner the eutectic structure of silver-copper alloys, was not so satisfactory with single-phase specimens, being less effective than chemical etching in this respect. Recent work by McCutcheon[64, 65, 66] has shown that when the correct experimental conditions are observed the process of cathodic etching has great possibilities, however.

Flow-lines in deformed metal can be shown up with a sharpness and clarity exceeding that attainable by chemical etching. Cathodic bombardment can reveal not only the usual microstructures of single and polyphase alloys, but also the extremely thin superficial deformed structures produced by the burnishing treatments frequently employed in the automobile industry. A very obvious advantage enjoyed by cathodic over chemical etching becomes apparent in the metallography of the noble metals. Although these metals are in general extremely resistant to chemical attack, they sputter with the utmost facility. Difficulties frequently caused by the partial decomposition of organic mounting materials in corrosive etchants can therefore be easily avoided.

The rate at which metal is removed from the cathode, being directly related to the current flow, is readily controlled. Grivet[67] has therefore recommended the employment of cathodic sputtering for etching specimens from which replicas are to be taken for subsequent examination under the electron microscope. The type of etch suitable for revealing structures under the optical microscope is not usually satisfactory for specimens intended for the electron microscope, and complete control over the etching process is, in the latter instance, virtually essential. Cathodic etching facilitates the attainment of this control. Details of the techniques and apparatus required have been described by Holland and Sutherland.[68]

Until the general introduction of metal evaporation in the middle thirties, cathodic sputtering was recognised as the most satisfactory method for producing mirrors of high reflectivity for scientific purposes. The techniques adopted have been described by Strong,[69] and in a more recent review of this subject Tolansky[70] discussed the characteristics of high reflectivity sputtered films of silver for multiple-beam interferometric purposes. For this type of work he concludes that good mirrors are attained by evaporation more readily than by sputtering. Providing sufficient attention is paid to the purity of the system, however, excellent results can be obtained by sputtering. Romanowa, Rubzow and Pokrowsky[71] reported transmission and absorption figures for sputtered mirrors better than any which have so far been reported for evaporated deposits.

Industrial Applications

Sputtering is a relatively slow process and cannot therefore compete with metal evaporation on routine applications where thick deposits have to be produced economically. Where the process involved is one which requires critical control of film thickness, the slow rate of deposition becomes something of an advantage, however. This advantage becomes even more pronounced in those applications where careful and precise adjustment of the electrical and optical properties of the deposit is involved. Sputtered films are more stable than those produced by evaporation: the physical properties of the latter change after deposition to such an extent that deposits to a definite specification are difficult to produce consistently by this method. Where accurate and consistent deposits are essential, as for example in the routine production of barrier layer photocells, sputtering becomes an essential process.[72-74]

The surface receiving a sputtered deposit heats-up. This fact restricts the choice of surfaces upon which sputtered films can be deposited to glasses, metals and stable inorganic compounds of relatively high melting point. Much fundamental work for which the process would otherwise be ideally suited has for this reason to be undertaken by evaporation methods.

The mechanism of sputtering does not depend directly upon the vapour pressure of the metal to be deposited, and many volatile metals sputter comparatively slowly. Occasionally the vapour pressure of the metal from which a deposit is required is so low that the required temperature cannot be attained in an evaporation unit. In such instances sputtering can often be profitably employed.

Details of the rate of sputtering under industrial conditions were published by Hulbert[75] as early as 1934. Information relating to more recent practice has been given by Fisher and Weber[76] in 1952, Umblia in 1951[73] and Bacquet in 1950.[77] Holland has[74] recently published the following empirical formula for calculating the rate of sputtering when large plane cathodes with negligible edge losses are employed :—

$$S = \frac{(V_a - V_o)\, i\, K}{dp}$$

Where S = deposition rate (mass deposited per second),
V_a = applied voltage,
V_o = constant (approximately 500 V.),
i = current density in mA./sq. cm.,
d = the distance separating the cathode from the receiving surface,
p = the pressure of the discharge gas at constant temperature, and
K = a constant, depending upon cathode material, discharge gas, and cathode arrangement.

Chemical Sputtering

Chemical processes are stimulated in the gaseous discharge, and the incidental reactions so caused used to be considered as major disadvantages of the sputtering technique. Pohl and Pringsheim[78] referred specifically to these chemical effects as early as 1912, and more recent workers have discussed the processes involved in somewhat greater detail. Tolansky,[79] for example, emphasised that the presence of organic impurities in the sputtering chamber could have catastrophic effects on the production of silver mirrors, leading to the deposition of dark brown films entirely useless for their original purpose. This organic contamination could last for a considerable time and might even lead under bad conditions to abandonment of the equipment. Traces of oxygen reduce considerably the quality of silver mirrors produced in a hydrogen atmosphere, and it was generally recognised at a very early date that many of the characteristic properties of sputtered deposits were due to the fact that they consisted, not necessarily of pure metals, but more probably of metallic compounds.

The earliest systematic investigation of the chemical effects induced by sputtering was probably that of Güntherschulze.[79] In this research the sputtering behaviour, in hydrogen, of twenty-four elements was studied. Many of these elements exhibited peculiarities which were attributed to electrochemical processes. The anomalies were explained by the behaviour of gaseous hydrides, the molecules of which diffused throughout the chamber without being deposited. Collision between these molecules and ions resulted in disintegration. The metal gas thus liberated condensed on the nearest cold wall, where ion bombardment rapidly turned it into a hydride again.

Chemical reactions were also found to have considerable influence upon the rate at which metal atoms were discharged from the cathode. In Meyers' investigation of 1931[54] rather interesting results were obtained. This study was carried out in mercury vapour at 1,000 V. The metals aluminium, silicon, manganese, iron, zirconium, molybdenum and tungsten all sputtered under these conditions at a constant rate of 1·08 atoms per incident ion. Nickel, beryllium, cobalt, chromium, platinum, palladium, copper and silver, on the other hand, sputtered at rates which ranged from 1·10–5·10 atoms per ion, the amount increasing directly with the tendency towards amalgamation. Methfessel[59] postulated chemical reaction as an essential stage in any sputtering process. In the initial stages of a glow discharge, the current density and cathode fall assume a relationship which is uniquely related to the nature and pressure of the gas present, but is independent of the value of the current and of the applied voltage. Disintegration of the cathode begins at this stage as a chemical process. The gas ions, which have been highly activated in the dark space, enter into unstable combination with the metal of the cathode. When these unstable molecules reach the luminous portion of the discharge, dissociation occurs and the metal vapour thus released condenses on the nearest cold wall. As the current increases, however, chemical sputtering becomes less important and is superseded by the normal sputtering mechanism at densities comparable to those employed for normal deposition work.

The chemical reactions possible are of course determined by the gaseous atmosphere employed. With oxygen and a platinum cathode platinum oxide can be readily deposited. Very stable oxides such as those of aluminium and beryllium can readily be deposited, even under such comparatively clean working conditions as those normally existing in an electron diffraction camera.[80]

Reactive Sputtering

This term was invented by Veszi,[81] and refers to a technique which deliberately exploits the tendency towards compound formation, particularly in the field of oxide deposition. For many optical purposes, non-absorbing layers of high refractive index are required. The choice of metallic compounds suitable for this purpose was hitherto limited to those which could be readily deposited, without decomposition, by thermal evaporation under high vacuum conditions. Holland[82] has emphasised that sulphides and chlorides, in particular, when used for such purposes yield deposits which scratch easily and are attacked by humid atmospheres. Those oxides which have the required optical properties and are also hard and chemically inert, usually have such a low vapour pressure that they cannot be readily deposited by thermal evaporation.

Reactions between metallic oxides and the heater material are accelerated under high vacuum conditions, with the result that the heater becomes embrittled, and the deposit obtained is actually a mixture of metal and oxide. At the high temperatures which would be re-

quired for the thermal evaporation of alumina *in vacuo*, reactions between the tungsten and oxide proceed rapidly. A compound such as titania, on the other hand, breaks down under high-temperature/low-pressure conditions to form the more stable lower oxide. Cadmium oxide decomposes completely under high vacuum conditions at 700° C. Thoria and zirconia are reduced by tungsten under high vacuum conditions at temperatures above 2,000° C.[83]

Stable films of most metallic oxides can be readily produced, however, by the simple expedient of sputtering the metal in an oxidising atmosphere. The techniques essential to the process were first developed in the laboratories of W. C. Heraeus during the war years, and details of this work are given in papers by Heisinger and König.[84, 85] A comprehensive summary by the same authors is given in the Heraeus Festschrift 1951. The authors refer here to the deposition and properties of films of aluminium, iron, cadmium, titanium, silicon and thorium oxides.

Holland's more recent researches[82, 86] have shown that three distinct mechanisms are involved in the process :—

(1) Due to the accelerating influence of the ionised gas an oxide layer is initially formed on the cathode surface. This oxide layer is removed in molecular form by the positive ion bombardment.

(2) The metal is emitted as a neutral gas vapour from the cathode and reacts with the oxygen molecules with which it comes into contact before being deposited on the receiving surface.

(3) The sputtered metal condenses to form a metallic layer which is subsequently oxidised by the direct absorption of gas molecules.

In many instances these mechanisms probably act simultaneously. Depending upon the circumstances, however, one or other is usually predominant. Cadmium oxide films have been prepared, for example, by simply using a layer of oxide on the cathode surface without any oxygen in the atmosphere.[87] It has also been observed that the low sputtering rate of aluminium is largely due to the presence of an oxide film.[53] Aluminium or magnesium electrodes may be operated for long periods under heavy loads in pure argon or neon with no sign of sputtering. Rapid sputtering begins in a matter of minutes and soon covers the inside of the apparatus with a metal deposit. The effect can be avoided by intentionally adding a trace of oxygen or water vapour to the inert gas.

Where an excess of metal is required to increase the electrical conductivity of the deposit, it is often convenient to dilute the oxidising atmosphere with argon. Sputtering conditions affect the conductivity of the deposit largely because they determine its purity and degree of oxidation. Holland and Siddall have stressed the need for close control over the oxygen content of the atmosphere employed. It is essential, therefore, to eliminate unknown quantities such as the gases desorbed from the interior of the apparatus before starting an experiment. The purity of the cathode material may affect the conductivity of the sputtered layer. This effect is discussed by Aitchison.[88]

Greatly increased sputtering rates can be achieved at large gas dilutions. The quantity of active gas required in argon can frequently be reduced to 5%. These quantities become comparable to the amount of extraneous gas liberated from the walls of a large metal system. In order to obtain satisfactory control under such conditions, it becomes almost essential to employ pumping equipment having a high throughput at the operating pressure. Booster pumps have suitable characteristics at the intermediate pressures used for industrial sputtering.[82, 83]

METAL EVAPORATION

Thin metallic films are extensively produced in industry by vacuum evaporation processes. These operations are pursued on a scale which is rapidly increasing, and the production of thin films by vacuum evaporation probably represents the greatest single industrial application of low pressure metallurgy. The process is spectacular and has been well publicised : much popular and technical literature has been based on the subject, and the reports of recent conferences and symposia devoted to evaporated films are comprehensive and rather formidable additions to a rapidly expanding pool of information. A recent book by a noted authority[89] has treated the subject in such a thorough and comprehensive manner that it would be pointless in the present instance to attempt more than a brief historical introduction.

The basic mechanism of vacuum evaporation is much simpler than that of cathodic sputtering. A small sample of the metal or compound is simply heated under high vacuum conditions until evaporation occurs. The metal vapour is then condensed at a cold receiving surface upon which the desired film is built up. An essential feature of the process is that it can only be carried out at pressures so low that the mean free path of the residual gas in the system is comparable to or greater than the distance of the evaporation source from the surface upon which a deposit is required. Under these conditions the evaporation source can be considered to emit atomic or molecular rays in all directions. These evaporated metal rays obey the normal laws of geometrical optics, the intensity of deposition being inversely proportional to the square of the distance from the source to the receiving surface and to the sine of the angle of incidence. By using a suitable pinhole system, intensity images of the source can be produced at any desired magnification. The latter technique has been employed during investigations into the performance of various types of heater.

Pohl and Pringsheim[78] studied the process in 1912 and appear to have been the first investigators to appreciate the advantages it enjoyed over cathodic sputtering as a technique for the deposition of pure metals. They stated " Kathodenzerstaubung und anodische Verdampfung bieten die Schwierigkeit, dass sich die Metallmolekule an der Stromleitung beteiligen und dadurch chemische Reaktionen zwischen dem Metall und dem Gasinhalt des Entladunggsrohres gegunstigt werden."

Because of the vacuum requirements, it was inevitable that interest in the evaporation technique developed only after the general introduction of the diffusion pump. The main experimental difficulties then lay in the development of a suitable type of evaporation source, evolution of sound vacuum engineering techniques, and recognition of the necessity for high speed pumping equipment.

Pringsheim and Pohl had distilled their metals from a magnesia crucible, but this method was found to be unsuitable for routine work. Refractory crucibles have a low thermal conductivity and poor resistance to

thermal shock. They tend to crack readily, and at high temperatures react with either the metal being evaporated or with the heater material. The metal being evaporated usually wets the crucible and in many instances diffuses through it, thereby leading to automatic crucible breakage on cooling. Perhaps the greatest disadvantage lies in the fact that the material of the crucible usually has an appreciable vapour pressure at the evaporation temperatures employed, with the result that the films deposited are badly contaminated. This defect is aggravated by gas evolution from the crucible which tends to react chemically with the evaporated metal.

Ritschl utilised the evaporation process in 1928 for producing silver interferometer mirrors,[90] employing a bare tungsten coil as a heater. Tolansky, who was at that time a guest research student in Berlin, worked with Ritschl and studied the technique which had been evolved. A description of the process, now largely of historic interest, was given by him in a recent paper.[70]

The system employed by Ritschl was evacuated by a four stage mercury diffusion pump provided with an efficient liquid air trap. The vacuum chamber was constructed of brass and the best mirrors were produced when the interior of this chamber was lined with plates and tubes of glass. The surfaces to be coated were hung about 30 cm. away from the tungsten filament, which had a platinum spiral loosely wrapped around it. The silver was prefused into the tungsten spiral before evaporation commenced. The object of the platinum was to retard the tendency of the molten silver to drop off the tungsten spiral. Thorough cleaning of the glass receiving surface was found to be an essential pre-deposition operation. Ionic bombardment was employed, a Rhumkorff coil being utilised for this purpose. Evaporation was commenced when a hard black vacuum had been achieved in the chamber.

An interesting feature of the Ritschl technique was the method adopted for fixing the silver film after deposition. A glass stopper having upon it a few drops of aqua regia was held below the mirror. A few seconds of such treatment increased the mirror transparency considerably. The film was then preserved and hardened by holding it for 1 minute over a crucible of warm hydrogen peroxide. Tolansky emphasised that this technique is only effective on mirrors which have been produced in equipment identical with that employed by Ritschl. The efficiency of the aqua regia treatment may have been associated with the presence of evaporated platinum in the film.

Telescope Mirrors

Cartwright and Strong surveyed the potentialities of the evaporation process in 1931.[91] A bare tungsten heater from which the metal was evaporated was found to be generally suitable except for the metals beryllium and aluminium, which dissolved the coil. Aluminium was, in fact, the most important metal to be considered, as the primary object of Strongs' research work had been to develop a method for aluminising large telescope mirrors. Further experiments were therefore carried out by Cartwright[92] in which crucibles of sapphire, graphite, magnesia, and thoria were employed. All these tests were unsuccessful because of metal-crucible reactions.

Complete success was finally achieved in a very simple manner. The limiting solubility of tungsten in aluminium was found to be only 3% by volume.[93] When a small quantity of aluminium was melted on a tungsten coil it could therefore absorb only a limited amount of tungsten before becoming saturated. If the tungsten coil was of large diameter wire, the amount dissolved from its outer surface did not seriously affect its physical or mechanical properties. This type of evaporation source is now almost universally employed. Considerable ingenuity has been directed towards the development of tungsten heaters capable of holding and evaporating large quantities of aluminium.[94,95]

The dimensions of the telescope mirrors handled meant that evaporation plants had to be of large capacity, and there is little doubt that some of the units built for astronomical work during the immediate pre-war period were the first examples of industrial scale vacuum equipment capable of being regularly operated at pressures of the order of a tenth of a micron.[96] The vacuum techniques evolved during this period were of considerable value during later atomic energy developments.

The success of vacuum metallisation led to developments which lie perhaps outside the scope of this survey, since they were concerned with the deposition of fluoride films intended to reduce the reflection of light from lens surfaces.[17] The process, known as blooming, is of importance in the present connection, however, because it marked an important stage in the development of vacuum evaporation techniques which were thereby introduced to a broad section of industry which would not have been interested in processes applicable only to astronomical work.

In the early days of the process many workers considered that oil pumps did not give a clean enough vacuum, and that for the attainment of really high quality deposits mercury diffusion pumps fitted with liquid air traps were required. Modern oil pumps fitted with efficient baffles permit good deposits of most metals to be readily obtained, and only on very high grade optical work can the difference between deposits produced in oil and mercury pumped systems be distinguished. Tolansky[70] observed, for example, that the reflectivities of silver interferometer mirrors produced by both techniques did not differ significantly. Only in the matter of absorption did the mercury product have superior properties. In few instances it is possible to attribute defects directly to the presence of oil vapour, as slight differences in the geometry of the evaporation source can lead to considerable differences in the rate of evaporation of impurities from the filament. Several instances quoted in the literature illustrate the difficulty of obtaining comparable conditions with both oil and mercury diffusion pump units, and therefore of obtaining a direct comparison of the film qualities possible by the two methods.

Film quality is directly related to the cleanliness of the receiving surface before the commencement of evaporation. Aluminium deposited on an inadequately cleaned surface has a poor adhesion and small blisters develop in it after a short period. Details of several preliminary cleaning treatments have been given by Strong.[69] The glass surfaces after such treatment are usually subjected to a glow discharge *in vacuo*. This discharge removes thin contaminating films from the specimens by ion bombardment, and also improves the cleanliness of the vacuum chamber by accelerating the degassing of glass and metal surfaces. In the old days, when pumping speeds were low, this glow discharge treatment could be carried out during the normal

pumping down process before the final degree of vacuum was obtained. Evacuation rates are now so rapid that such a technique becomes impractible.

The system is first pumped down hard so as to remove as many contaminating films as possible. Controlled quantities of a suitable gas are then admitted and the cathodic bombardment carried out. Strong floods the system several times with oxygen as this assists in removing the contamination. Holland[74] has described the precautions required to prevent the glow discharge from entering the diffusion pump baffle and causing partial decomposition of the oil present. Unwanted sputtered films near the mirror surface must also be avoided.

Where metal substrates have to be cleaned, they are usually directly connected to one side of the high tension supply, which should preferably be rectified. A suitable discharge gas in such cases is hydrogen. The adhesion of aluminium films to glass, however, is poor if a reducing or inert rather than an oxidising gas is used for cleaning the substrate. Dunoyer[98] has used electron bombardment as a method for removing surface contamination. The electron source is a hot cathode and the fixture used for holding the receiving surface is made the anode.

Industrial Evaporation Processes

Developments made during and after the war in the field of metal evaporation have been mainly those required to transform a highly skilled laboratory technique into an industrial process. Automatic and continuous coating equipment has been introduced and the necessity for high speed pumping equipment adequately recognised. The equipment now employed is robust and capable of being satisfactorily handled by unskilled labour having no understanding of the principles involved.

Automatic equipment is employed for depositing aluminium on to the fluorescent screens of cathode ray tubes which have a very high light output,[99] and similar techniques are employed for producing the reflecting coating required on the interior of lacquered headlamp mirrors. The techniques involved in large scale metallisation of plastic mouldings have been described in detail by Bateson[100] and more recently by Holland.[89] Large volumes of gas are evolved from most mouldings, particularly those containing plasticisers of low boiling point, and backing pumps required for evaporation units engaged on this type of work have therefore to be of greater capacity than those used for optical purposes. Deposition times are kept down to seconds in order to minimise the outgassing tendencies of the plastic, and this requirement means that evaporation sources must be capable of evolving metal at very high rates. A substrate with a craze-free surface is essential for the attainment of bright metallic finishes, so that lacquering techniques play an important part in decorative metallisation operations. Evaporation times usually represent, therefore, only a small proportion of the total processing cycle.

Plastic sheeting in roll form can be metallised by vacuum evaporation. The volatile materials evolved from the sheet usually necessitate the use of booster pumps and refrigerated traps to prevent contamination of the rotary pump oil. Paul Alexander[101] has, since 1937, designed and produced several machines used for plastic sheet coating, and equipment for the continuous coating of paper condenser plates with zinc is now extensively employed. The high vapour pressure of the liquid zinc makes it possible for coating rates to exceed several hundred feet per minute.[102] Much water vapour is evolved in the vacuum chamber when the paper is heated in its passage past the evaporation source, and the most satisfactory method of maintaining good vacuum conditions in this type of plant appears to be the installation of a retractable liquid air trap which can be inserted into the chamber close to the surface of the heated paper.

(to be continued)

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Jamaican Bauxite Developments

THE Ewarton plant of Alumina Jamaica, Ltd., will shortly come into production on a small scale says a Barclays Bank D.C.O. message from Jamaica. As the plant settles down a steadily increasing rate of production is expected by the middle of next year. If all goes well, and market conditions are favourable, it is anticipated that full production at the rated capacity of 250,000 tons of alumina per annum will be achieved in the latter part of 1961. The Aluminum Company of America is at present prospecting for bauxite in the Clarendon area. They expect to continue their testing for some time before deciding whether to start mining operations.

Zone Melting of Boron Facilitated

New Technique for Pressing Rods

THE discovery of a method for pressing rods of commercially available powdered boron has led in turn to successful floating zone melting of that extremely high melting point element. The technique will provide larger crystals for basic research studies of the semiconductor than have been available before. A paper describing the new method was presented by Dr. E. S. Greiner of Bell Telephone Laboratories to the Metallurgical Society of the A.I.M.E. in Chicago recently.

Because of its physical properties, boron does not lend itself to floating zone melting, since it is not possible to form the powder into a suitable shape by pressure alone. The pressed forms are so friable that they crumble as soon as any attempt is made to remove them from the forming dies.

In one aspect of Dr. Greiner's new technique, boron powder, −20–100 mesh in size, is placed in boiling boric acid solution, and the mixture boiled to dryness. This coats the boron granules with boric acid, and the powder can then be pressed into forms which can be handled without breaking.

The formed bars, or compacts, are heated in two stages under vacuum for one hour, at temperatures of 300°–600° C., to decompose the boric acid into boron oxide. This compound forms a liquid coating on the boron particles. When the compact is cooled, this liquid hardens, and bonds the powder into a strong bar.

The bar of boron powder is mounted vertically for floating zone melting, and a high frequency induction coil is used to melt a narrow band or zone of the material. The molten zone is moved down the rod by raising the bar through the coil, while the melted boron freezes or crystallises above the coil. During this operation, the boron oxide binder sublimes before the melting point of boron (about 2,000° C.) is reached. Crystals as large as 0·1 in. were obtained, many of which were twinned.

The apparatus used in the floating zone refining is similar to that used in the treatment of other materials, except that the rod of boron is enclosed within two concentric transparent quartz tubes. The inner tube is slotted; when the subliming boron oxide coats the tubes enough to restrict vision, the slotted tube is rotated to give a clear field again. This modification to the equipment has previously been used in the floating zone melting of gallium arsenide at the Bell Laboratories by J. M. Whelan and G. H. Wheatley.

Dr. Greiner watches a rod of boron powder being zone melted.

Preparatory to mounting the compact, the corners of the square bar are ground off, to minimise the "cage" effect, in which the corners do not melt. Also, in order to lower the resistivity of the boron, to allow it to be heated and melted by high frequency induction, the top end of the bar is mounted in a graphite cup, which can be readily heated by such means.

Canadian Refractory Orders

WITH an already existing world-wide export trade amounting to over £200,000 annually and covering some twenty-seven countries, The Marshall Refractories Group, Sheffield, are increasing the tempo of their activities in the export markets. At about the same time as Mr. Hale, Marshall's sales director, was making his successful five-week tour of Latin America last August, Mr. G. T. Wragg, Marshall's chief representative, was dealing personally with trade enquiries in Canada. As a result of this visit, orders have already been received to the value of £8,600 from Dominion Steel & Coal, Dominion Foundries & Steel and Canadian Steel Wheel. The Marshall Refractories Group has previously supplied carbon refractories and insulating bricks to Canadian companies through their local agents and representatives. However, the success of Mr. Wragg's visit has not only established that it is the personal contact which proves the most fruitful in negotiation, but has had the effect of inspiring their agents to contemplate the potential market which exists for British-made refractories.

Burton, Griffiths and Delapena

BURTON, GRIFFITHS & CO., LTD., Kitts Green, Birmingham, and Delapena & Son, Ltd., Cheltenham, announce that they have joined forces for the distribution of Delapena honing machines and equipment in the U.K. (including Northern Ireland). This arrangement has been made with the object of offering maximum service to users of honing equipment.

Metallurgical Examination of a Failed Electro-Magnetic Liquid Metal Pump Channel

By G. H. Broomfield

Atomic Energy Research Establishment, Harwell

Examination of a 13% chromium steel electromagnetic liquid metal pump channel, which failed by cracking after some 950 hours rig testing, showed that the fracture was caused by fatigue stresses, probably imposed by pressure pulses in the liquid metal. The evidence indicated that liquid metal corrosion had some influence in promoting failure.

LOW melting point metals in the liquid state are very efficient heat transfer media, and they have been used in this role in fast neutron[1, 2] and thermal neutron[3] nuclear reactors. A liquid metal has also been proposed as a fuel solvent in a thermal neutron reactor,[4] and for this purpose bismuth has the most favourable nuclear[5] and physical properties,[6] together with reasonably satisfactory metallurgical properties with respect to compatibility with container materials and capacity for dissolving a nuclear fuel such as ^{233}uranium.[7]

As part of a programme of feasibility and design studies of possible liquid metal fuelled reactors, the technology of circulating bismuth systems has been studied in several laboratories. One particular aspect of these studies has been that of the electromagnetic pumps[8] which may be used to circulate the fuel or coolant. The operation of such pumps depends on the influence of a magnetic field on the liquid metal, which acts as a conductor for a heavy electric current: the arrangement of the channel, field poles and connections for a simple D.C. conduction pump is shown in Fig. 1.

Pump channels for bismuth are best made of ferritic low-nickel steel, and since both the current and the magnetic field in the liquid metal pass through the steel, the walls must be thin to reduce by-pass losses. The channels may therefore be highly stressed. The preference for ferritic steels for bismuth systems in general, and pump channels in particular, is due to the relatively high solubility of nickel and manganese in bismuth, making austenitic steels unsuitable. Operating temperatures may be as high as 550° C., but the more refractory metals are not suitable, either because of their relatively high solubility in bismuth or their poor oxidation properties.

Iron has a low solubility in bismuth: it is about 4 p.p.m. at 400° C., and 25 p.p.m. at 550° C.[7] This variation of solubility with temperature can, in systems in which bismuth circulates through a temperature gradient, lead to corrosion by the mass transfer mechanism which depends on solution of iron from the hot part of the system followed by precipitation and trapping of iron crystals in the cold part: the liquid metal returning to the hot part is then unsaturated as its temperature increases, allowing solution of more iron. Mass transfer corrosion can be inhibited by first de-oxidising the system by adding 500–1,000 p.p.m. of magnesium to the bismuth and then adding 250–500 p.p.m. of zirconium (this figure depending on the solubility of zirconium at the minimum circuit temperature). Zirconium in solution in bismuth combines with nitrogen in the steel to form an effective solution barrier of ZrN[7] a few hundred angstroms in thickness. The efficiency of this inhibition technique depends on the continued availability of nitrogen from the steel: a very low nitrogen content or a high level of alloying elements capable of forming strong nitrides may prevent ZrN film formation or lead to its early decay.[9]

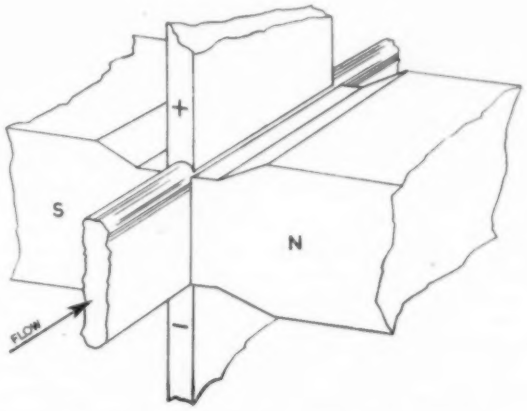

Fig. 1.—The arrangement of channel, connections and field poles in a simple D.C. conduction pump.

Bismuth expands on freezing, leading to a requirement for very positive directional solidification to a free surface if a rig is to be shut down without first draining the liquid metal. For this reason lead-bismuth eutectic, which contracts on freezing,[10] is sometimes used for preliminary rig work, and although it dissolves less zirconium it can be inhibited by the same technique as that used for bismuth.

Pump Details

The pump channel which forms the subject of the present investigations was taken from a loop in which lead-bismuth had been circulated for the purpose of examining the effect of inhibitors on pump efficiency.[11] The channel was part of a 2-phase (separately excited) A.C. conduction liquid metal pump designed to pump 15 gallons of bismuth per minute at 15 lb./sq. in. pressure,[12] and had been manufactured from 0·036 in. wall thickness, 13% chromium steel tube with copper conductors brazed to it with Johnson Matthey G.6 silver brazing alloy. It was a replacement for the original pump

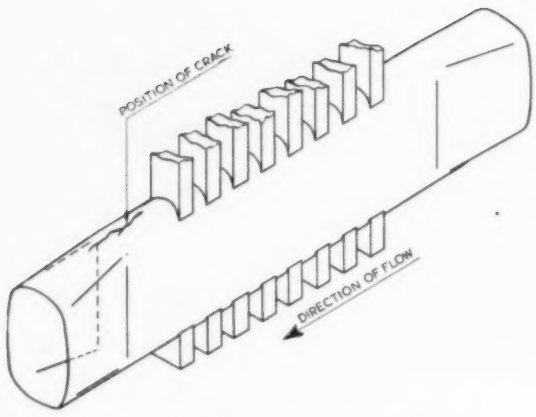

Fig. 2.—Diagram of the pump channel.

channel which had failed by perforation due to corrosion by bismuth. The pump was used to pump the lead-bismuth eutectic alloy in a nominally isothermal elliptical loop, and failure in this instance was due to a crack which developed in the position indicated in Fig. 2 and allowed liquid metal to leak out.

Rig Life of the Channel

Total rig life of the pump channel was approximately 950 hours, during which time the pump was operated at various power inputs and at temperatures between 350° and 400° C. The pump operated at full power for a total of about ten hours, at various settings between 20% and full power for periods totalling approximately 250 hours, and for the remainder of the time at less than 20% of full power. The pressure rise through the pump when operating on full power was 7 lb. sq. in., and the total pressure in the liquid metal was augmented by a static head of approximately 9 lb. sq. in. Excessive vibration, possibly due to loose core irons, prevented full power operation near the end of the period.

For the first few hundred hours the liquid metal contained no oxygen getting or inhibiting addition. Magnesium was added first, to a concentration of approximately 400 p.p.m., and this coincided with a reduction in pump efficiency. Zirconium was added, after a total of about 750 hours running, to a concentration of about 100 p.p.m. This was followed by a period in which pump performance improved and which terminated with the failure.

General Examination of the Channel

The crack, which was at the outlet end of the pumping region, was clearly visible on the outside. It was branched and was not associated with any appreciable distortion of the tube. A bend test showed that the steel had not been embrittled by its service at high temperature, and examination with penetrating crack-detecting fluid revealed no further cracks. Lead-bismuth alloy which had penetrated the crack made it readily discernible in X-radiographs but no further defects were revealed.

The pump channel was not completely wetted by the lead-bismuth alloy, but was almost so (about 80%) in the region of the crack, and on much of the portion of tube to which the electrodes were joined. Examination of the inside of the tube by electron diffraction revealed no zirconium nitride layer.

Analysis

An analysis of the steel gave the following results:

Chromium	13·1%
Manganese	0·15%
Nickel	0·22%
Carbon	0·12%

These values are normal for the type of steel, although the nickel content is high for this type of application.

The Fracture Surface

The fracture surface revealed when the crack was parted showed two features typical of fractures induced by fatigue: it had a two-zone structure, with the finer textured zone towards the inside of the tube, and the fracture was stepped, the steps tapering away from the inside of the tube (Fig. 3). These features show that the crack started at the inside of the tube and slowly progressed outward.

Micro Examination

The etched structure of the steel was typical of the type with the thermal history of the pump tube. The region of the crack was not plastically deformed (Fig. 4). Corrosion pitting was observed near the crack and near the flat sides of the channel a few millimetres from the crack. No defects due to steel manufacture or tube fabrication were detected.

Stress in the Region of the Crack

Stresses in the pump channel in the region where the crack occurred were not calculated by the designers, since the original design incorporated a channel of different form, but an indication of the stress level due to the internal pressure of liquid metal can be obtained by using a method described by Brown[13] for calculation of stresses due to internal pressure in thin elliptic tubes. Such pressure results in a bending moment in the wall at the ends of the major axis of the tube, which is related to the dimensions of the tube and the internal pressure by the expression

$$M = \frac{pD^2}{12}\left[1-\tfrac{1}{2}\left(\frac{d}{D}\right)^2\left(\frac{K}{E}+1\right)\right]$$

in which M is the bending moment, p the internal pressure, D and d the major and minor axes; K and E are elliptic integrals.

The values of D and d for the pump channel were approximately 1·8 in. and 0·4 in., and a graph by

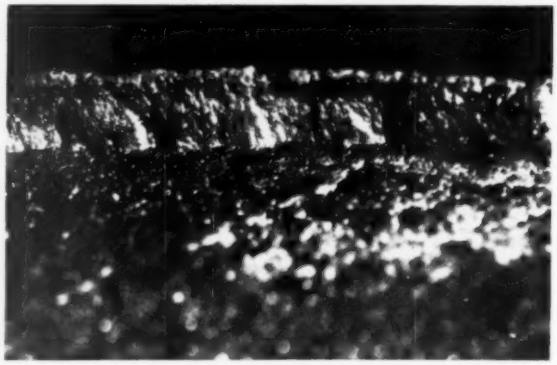

Fig. 3.—The fracture surface, showing a two-zone structure and tapered steps. × 15

Brown gives, for this axis ratio, a value of 0·88 equal to the term

$$\left[1-\tfrac{1}{2}\left(\frac{d}{D}\right)^2\left(\frac{K}{E}+1\right)\right]$$

The measured pressure rise through the pump was probably an R.M.S. value, hence the maximum internal pressure was $(9+7\sqrt{2})$ lb. sq. in. alternating with a minimum of 9 lb. /sq. in. at 2×50 c./s. Using the above formula, the bending moment varied between 2·135 and 4·49 lb. in.

The maximum fibre stress f at the inside of the tube is then given by

$$f=\frac{My}{2240\,I}\ \text{tons/sq. in.}$$

where y is half the thickness of the tube wall and I is one twelfth of the cube of the thickness of the tube wall. At the lower pressure, therefore, $f=4{\cdot}28$ tons/sq. in., and at the higher pressure $f=9{\cdot}27$ tons/sq. in. Since the pump channel section was not a true ellipse, and the ends of the region where the crack occurred were constrained by regions probably under lower stress the above values must be taken as approximations.

Discussion

Results of the metallurgical tests made on the cracked pump channel show that the crack was due to fatigue stresses which exerted a maximum tensile stress at the inside part of the tube affected. Since there are no indications of crack formation commencing at the outside of the tube, it is probable that the fatigue stresses were undirectional, corresponding with the fluctuating stresses produced by internal pressure pulses due to voltage peaks of the A.C. supply.

The magnitude of stress required to produce the crack is difficult to estimate from metallurgical considerations, in view of the inadequate amount of knowledge of the effect of liquid metal corrosion on fatigue. Fatigue under aqueous corrosion conditions leads to a reduction of endurance or stress limits to small fractions of their original values, and to the elimination of the "fatigue limit" effect in ferritic steel,[14] but anomalous results have been obtained from tests on metals in contact with liquid metals. In some instances, when corrosion by the liquid metal has occurred, there has been no significant change in endurance;[15] but a more common effect appears to be that of a reduction of properties, which is sometimes more marked after long exposures at low stress.[16] It is conceivable that the degree of corrosion observed in the pump channel was sufficient to cause the apparently low stress operating (the fatigue strength of this type of steel in reverse bending should be of the order of $\pm$ 26 tons/sq. in. at 400° C.)[17] to initiate a crack, but this cannot be confirmed. If it is considered that the stress cycles which were effective in producing the crack were those developed in full power operation, the endurance was only $3{\cdot}6\times10^5$ cycles, which indicates a stress level well in advance of that which the material would have withstood for a long period.

Additions of magnesium and zirconium were made to this rig so that their influence on pump performance could be observed. They did not appear to have been effective in producing an inhibiting layer of zirconium nitride, probably for the reason that the nitrogen in the steel had become fixed by combination with chromium or silicon during the period of operation prior to the addition of zirconium.

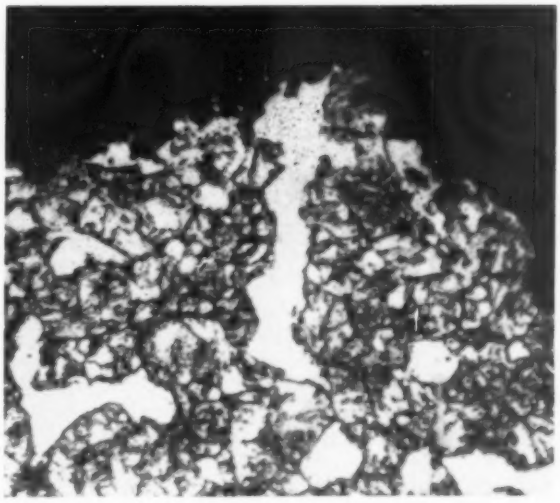

Fig. 4.—The structure at the fracture, showing no plastic deformation. × 800

Conclusion

The pump channel failure was a result of fatigue stress probably imposed by pressure pulses in the liquid metal. The evidence indicated that liquid metal corrosion had some influence in promoting the failure.

Acknowledgments

Thanks are due to Messrs. R. E. Derbyshire, A. Parker and D. Wilkinson all of the U.K.A.E.A., for radiographic, analytical and electron diffraction work, and to Dr. B. R. T. Frost for useful discussions.

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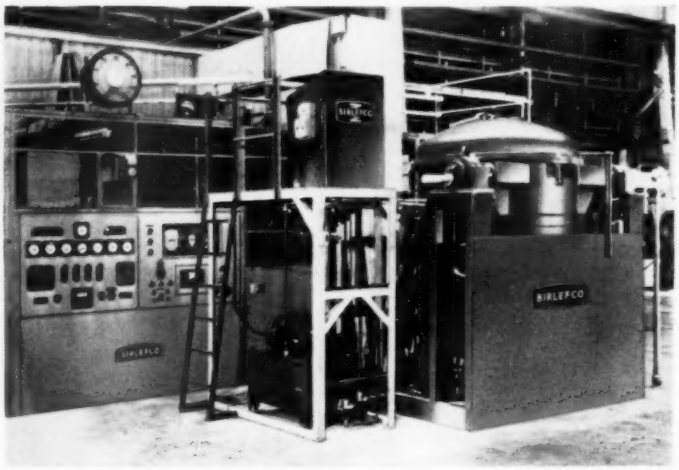
Low Cost Refining of Molten Metals

Birlefco Installation in Birmingham



THE vacuum melting furnace has proved an expensive but useful tool in the production of better alloys and of cleaner metals. Stream de-gassing equipment, alternatively, greatly reduces costs but is only suitable for relatively large batches of metal and has a limited effect on chemical analysis and, therefore, properties. In practice, it is limited in use to large forging ingots. Various ladle de-gassing systems are also in use, but are limited in the degree of de-gassing that can be obtained in a practical time, bearing in mind the difficulty of keeping metal hot in a ladle.

Birlefco (Birlec-Efco (Melting), Ltd.) have developed a de-gassing and vacuum refining unit which claims to overcome many of these criticisms. It is relatively inexpensive but gives close control over temperature and pressure. Turbulence of the metal in the vacuum vessel is induced electro-magnetically and cycle times are therefore reasonably short.

A unit of this type has recently been installed at the Birmingham Works of Henry Wiggin and Co., Ltd., for the vacuum refining of special alloys. Basically it is a 15 cwt. mains frequency induction melting furnace housed in a tiltable vacuum tank. A water-cooled copper induction coil, which is supported by a magnetically-shielded steel framework, forms the furnace proper. Special insulating techniques and careful design work have been necessary to eliminate the difficulties of handling high voltages in vacuum, thus ensuring that no troubles arise through electrical breakdowns within the furnace.

The choice of mains frequency was based, not only on the lower cost of the mains frequency electrical gear when compared with H.F. equipment, but also on the inherently greater degree of turbulence which a mains frequency furnace imparts to the molten metal. This turbulence stirs the bath thoroughly and brings new metal to the surface where gases and volatile metals can be drawn off. The present installation has a rating of 300 kW. and is arranged for connection to a low tension three-phase supply. This rating ensures ample power for melting, though, for refining service only, a maximum rating of 200 kW. would suffice.

The vacuum tank, which houses the furnace, is mounted on trunnions and tilted by twin hydraulic cylinders. The tank movement produces an approximately lip-axis pouring effect from the furnace, and a retractable bridge over the top flange of the tank carries the metal stream. Hydraulic controls open and close the vacuum tank lid, and pneumatic clamps lock the lid on to the tank. The furnace mouth carries a refractory cover, which reduces heat losses and cuts down the loss of metal by splashing. The cover has holes which correspond with a viewing port in the chamber lid.

The vacuum pumping lines are connected to the tank concentrically with the tilting trunnion, and in this way pumping connections are not demounted when the furnace tilts. The present installation includes a three-stage pump set which gives ample gas handling capacity over the whole range. Automatic controls maintain the correct pumping sequence and a dust trap protects the pump set from damage by fumes and metal splashes. Pirani and dial gauges monitor the furnace pressures.

The whole equipment was designed for use by relatively unskilled personnel with a minimum of technical supervision.

A typical vacuum refining cycle occupies about one hour. Liquid metal is brought to the furnace by ladle and, when the furnace is charged, the hydraulic gear lowers the lid into position over the chamber and pneumatic clamps lock it in place. The chamber pressure is then reduced to about 200-300 microns in about 15 minutes. During refining, sufficient power is applied to the furnace to ensure thorough stirring of the molten metal and to maintain the temperature at the desired value.

After vacuum treatment, air is re-admitted to the furnace, the hydraulic gear raises the lid, and the retractable spout is placed in position. The temperature of the metal is adjusted and checked by dip thermocouple and various other checks are made, after which the furnace tilts, pouring the vacuum refined metal into a ladle for casting or teeming in the conventional way.

The application of vacuum to the melt removes gases and volatile metals. The results obtained so far have proved extremely satisfying and the high temperature properties obtained on vacuum-refined metals have exceeded those forecast, basing the estimates on work done in the laboratory. A standard basic refractory

(*continued on page* 211)

Silver-Aluminium Alloys for Brazing Titanium and its Alloys[1]

By H. C. Davis, B.Sc., B.Sc.Eng., A.R.I.C.

Metallurgy Department, Royal Aircraft Establishment

The development of two solders suitable for brazing titanium to itself and to Ti–6% Al–4% V and Ti–5% Al–2½% Sn alloys is described. The best results are obtained at 650–660° C. in vacuo or purified argon, with solders containing 69% Al–30% Ag and either 1% Ni or 1% Mn. Results are given of elevated temperature tensile tests on brazed joints, and of compression tests on sandwich panels with alloy faces and honeycomb and corrugated cores in titanium and titanium alloy. The brazed joints show some ductility, but suffer appreciable corrosion by sea-water spray.

THE development of high-speed, high-altitude aircraft and projectiles has directed much attention to the possible use of titanium and its alloys as media of construction, particularly the latter, due to their high strength/weight ratio. Assemblies have been fabricated by welding and by brazing, using pure silver or silver-rich alloys as solders at working temperatures of 1,000° C. or more. Such temperatures are sufficiently high to incur loss of properties of the heat treatable alloys, and in order to avoid this it is necessary to restrict the brazing temperature to below 700° C.

In sandwich constructions flux residues are undesirable, so that a fluxless process is to be preferred, and it was considered that brazing *in vacuo* or an inert atmosphere such as argon would be the approach most likely to prove successful. In view of the difficulty, if not impossibility, of purging all the air from within a honeycomb-cored sandwich by means of a flow of inert gas, removal of the air by pumping would be essential in such cases, and work was therefore confined mainly to brazing *in vacuo* with a few subsidiary experiments in argon.

De Cecco and Parks[2] and Lewis *et al*,[3] in experiments on torch and furnace-brazing titanium, found the best joints to be made with fine silver as filler, and that various brazing alloys gave less satisfactory joints due either to brittleness or to erosion of the titanium. Joints obtained using 2S aluminium or 61S aluminium alloy were found to be reasonably ductile.

Experimental

Experiments with commercial brazing alloys such as Easiflo showed these materials to be unsuitable for a vacuum process, since volatile constituents such as cadmium and zinc evaporated and left a high melting point residue. Tests at 680° C., with commercially pure aluminium gave promising results *in vacuo*, but wetting was imperfect, apparently due to fouling of the metal by gaseous residues, although the furnace was continuously evacuated to a pressure of approximately 1 micron. Greatly improved results were obtained by enclosing the test assembly in a loosely-fitting box of titanium which, when hot, acted as a " getter " and removed any harmful residues in the atmosphere or evolved from the walls of the furnace.

Form of Brazed Joint

The primary test of a sandwich structure is made by end-compression of the test panel, whereby the face plates tend to be forced away from the core and the joints fail largely in tension. In view of this, it was considered that strength values obtained from T-shaped test pieces would be more informative as to the probable strength of sandwich panels than would lap joints. A disadvantage of the 'T' specimen is that, where failure occurs through the joint, the cross-sectional area of failure cannot easily be measured, and failing stresses are consequently difficult to calculate. For this reason the strengths of the joints have been expressed as the failing load in lb. in. run of joint.

Method of Brazing

Joints were made between baseplates of 0·063 in. and vertical webs of 0·021–0·032 in. sheet material (nominal thickness). The base-plates and webs were mounted in a jig with a strip of solder foil placed between each web and base-plate. Small dead-weights were attached to the webs to ensure that they fell into close contact with the bases as the solder softened and melted. Two holes were drilled in the base-plates to locate them in the jig, and these same holes were used to attach the brazed specimen to a 'T'-shaped jig for mechanical testing. The base-plates measured 1 × ¾ in. and the webs 2 × ¾ in., so that a joint ¾ in. long with fillets on both sides of the web was made. The strips of solder were cut from foil 0·005 in. thick and measured ¾ × ⅜ in. One of the joints is shown in Fig. 1. In preparing the solders, all annealing operations were conducted in an argon atmosphere to minimise oxidation.

Prior to assembly in the jig, the titanium parts were first chemically cleaned by immersion in a bath containing 25% nitric acid and 2·5% hydrofluoric acid until effervescence had practically ceased. After removal from the bath, the pieces were washed, rinsed in methylated spirit and dried on a clean cloth. Care was taken to avoid touching the pieces with the bare fingers from the time they entered the cleaning bath until they were removed from the jig after brazing. The surface of the brazing solder was abraded with fine emery paper, and after the pieces had been cut to size they were rinsed in benzene and dried in air.

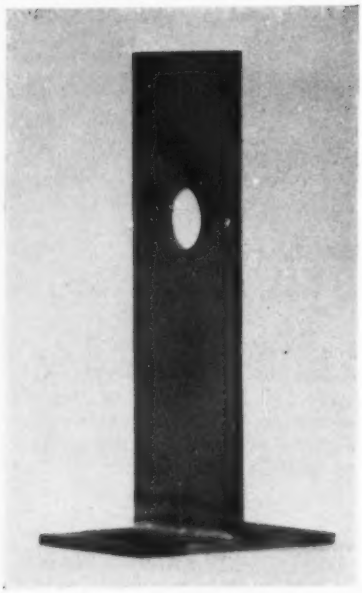


Fig. 1.—Brazed tensile 'T'-piece

After assembling in the jig, a thermocouple was attached so that the junction touched lightly against one of the webs about half an inch above the base-plate. The jig assembly was then placed in a chemically cleaned shroud, made from commercially pure titanium sheet, and placed in the vacuum-furnace (Fig. 2). Whilst evacuation was proceeding, the heating furnace was run back and the heating current adjusted so that the furnace reached a temperature of 700–750° C. by the time that the reaction tube was evacuated (pressure 0·5–1 micron in 20–30 minutes). At this stage the furnace was moved over the reaction tube and the specimens to be brazed were heated to 650° C. in 15–20 minutes. In general the temperature was held at 650°–660° C. for 10 minutes, but variations of both temperature and time were made during the investigation. At the end of the brazing period, the furnace was run back and the reaction tube and contents allowed to cool in air until the temperature had fallen to 500° C., or slightly less. An air blast was then turned on to cool the tube as rapidly as possible, and when the temperature of the test pieces had fallen to below 100° C., air was admitted to the tube and the contents removed.

Tensile Tests

The strength of joints made at 680° C. by the above procedure using aluminium as filler were very erratic, varying between 400 and 1,300 lb. in. run of joint. A more satisfactory solder was sought, having, if possible, a lower operating temperature than aluminium, and in view of the recognised good wetting properties of silver it was thought that alloys of silver and aluminium might prove useful.

A solder of the eutectic composition 72% Ag–28% Al (M.P. 558° C.) was found to have good wetting and fillet-forming properties and gave much more consistent strengths than aluminium alone. It proved of little practical value, however, since foil wider than about ½-in. could not be produced. Further tests with proportions varying between 50% Al–50% Ag and 90% Al–10% Ag all produced easily rollable alloys. The best joints were obtained with 70% Al–30% Ag at 650°–660° C., whilst good joints were obtained at 630°–690° C., inclusive.

Effect of Atmosphere

Tests were made to determine the minimum degree of evacuation necessary, and it was found that the presence of air at a pressure of 5 microns was sufficient to prevent joint formation, and subsequent work was carried out in the highest vacuum obtainable (0·5–1 micron). In an argon atmosphere in which the gas was supplied direct from a cylinder to give a pressure of 0·5–1 atmosphere, provided the titanium shroud was used, sound joints could be made although the shroud showed appreciable discolouration. Without the shroud the specimens suffered oxidation sufficient to inhibit brazing, and it is evident that if joints are to be made in argon some means must be provided to purify the gas.

Effect of Time and Temperature on Joint Strength

The influence of the time for which joints are held at the brazing temperature is shown in Table I. The results indicate that a time of about 10 min. is necessary, and that much longer or very short times are to be avoided. Table II shows the greatest strength to be obtained with a 10 minute brazing time at 650°–660° C.

Test on Sandwich Panels

Three sandwich panels were made and tested in end-compression. In all cases the face plates were of Ti–6% Al–4% V sheet (0·032 in. thick, nominal) and the 70% Al–30% Ag solder was used in the form of foil 0·005 in. thick. One panel had a corrugated core of commercially pure titanium formed from 0·021 in. thick sheet, the corrugations being equilateral triangles of 0·70 in. side. The other panels had honeycomb cores with cells measuring ½ in. across the flats and ½ in. deep, the core being made from 0·003 in. thick titanium strips eyeletted together to form the honeycomb. The largest panels which could be accommodated in the vacuum furnace measured 6 × 3 in. and components of this size

TABLE I.—EFFECT OF BRAZING TIME ON THE STRENGTH OF BRAZED JOINTS (Base: Ti–6% Al 4% V; Web: Ti; Solder: 70% Al–30% Ag)

Time at 650°–680° C. (min.)	Failing Load (lb./in. run)	Mean Value (lb./in. run)
1	595, 910	752
3	1200, 1010	1105
5	1190, 1162	1175
7	1050, 1410	1230
10	1400, 1556	1478
20	1055, 1055	1055
60	No joint: solder diffused into parent metals.	

TABLE II.—EFFECT OF BRAZING TEMPERATURE ON JOINT STRENGTH (Materials As In Table I)

Brazing Temperature (° C.)	Time at Temperature (min.)	Failing Load (lb./in. run)	Mean Value (lb./in. run)
630–640	10	932, 1185	1059
650–660	10	1400, 1556	1478
660–670	10	1253, 1387	1316
680–690	10	1388, 1215	1302
630–640	20	1035, 1355	1195
650–660	20	1055, 1055	1055

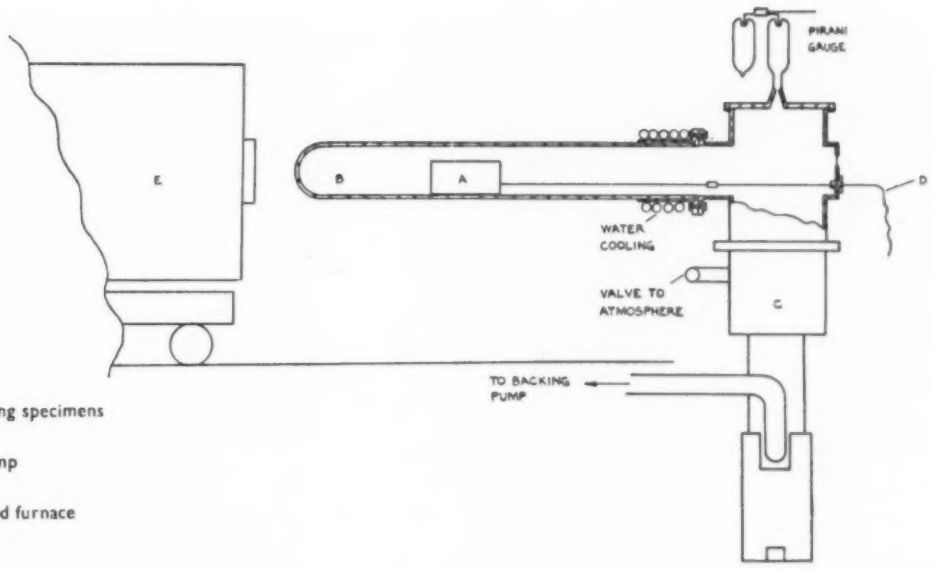


Fig. 2.—Vacuum brazing apparatus

A. Shroud containing specimens
B. Silica tube
C. Oil diffusion pump
D. Thermocouple
E. Resistance-heated furnace

were cut from the various materials and assembled in a channel-shaped shroud of titanium which enclosed the edges of the sandwich. Brazing was carried out for 10 minutes at 650°–660° C.

Prior to assembling the sandwich, the face plates and cores were chemically cleaned as for the tensile specimens. The brazing foil was cleaned by immersion for a few seconds in the nitric-hydrofluoric acid bath and removing the black film produced by a further immersion in nitric acid 15% v/v (approx.). The foil was then washed and dried.

The corrugated panel gave values of 37·2 and 36·8 tons sq. in. for the equivalent* and panel† stresses, respectively, and this is about the maximum that could be expected, since it is slightly greater than the 0·1% proof stress of the core material which contributes significantly to the strength of this type of panel. For the honeycomb panels, equivalent stresses of 44·3 and 30·8 tons sq. in. were obtained with corresponding panel stresses of 58·0 and 39·0 tons sq. in.

Ternary Solders

It became evident during the testing of these panels that the brazed joints were brittle since, at the point of failure, one or both of the faces stripped violently away from the cores. In attempts to produce more ductile joints, small additions of other elements were made to the binary solder, and for this purpose silicon, copper, nickel, manganese and titanium were tried. Of these, only nickel and manganese proved useful, the others all giving joints with strengths much inferior to those obtained with the binary alloy, without increased ductility. The amounts of nickel and manganese which could be added were limited to about 2% by weight at the expense of aluminium since, above this amount, the alloys became increasingly difficult to roll into foil. The tensile strengths of joints made with solders containing 2% of the ternary addition were not superior to those containing 1%, so that further tests were limited to the 1% ternary alloys. No benefit was obtained from a quaternary solder having 0·5% each of nickel and manganese. The solidus-liquidus range for the ternary 1% Ni and 1% Mn alloys was found to be similar to that of the binary alloy, so that the brazing temperature was unaffected by the ternary additions.

Some values for the strength of joints brazed for various times using the manganese and nickel ternary solders are given in Table III. These show that the greatest strength is obtained with a brazing time of 15 minutes for the 1% manganese solder and that little advantage is gained by prolonging the time beyond 10 minutes for the 1% nickel alloy. Similar results were obtained with joints in which the Ti–6% Al–4% V alloy was brazed to itself, and these times were therefore adopted as standard. A summary of the average strengths obtained with various parent metal combinations using these ternary solders is given in Table IV, from which it is evident that at room temperature, and with the exception of titanium brazed to Ti–6% Al–4% V

* *The equivalent stress* is determined by dividing the failing load by the cross-sectional area of a single plate of face material having the same surface dimensions and weight as the panel.

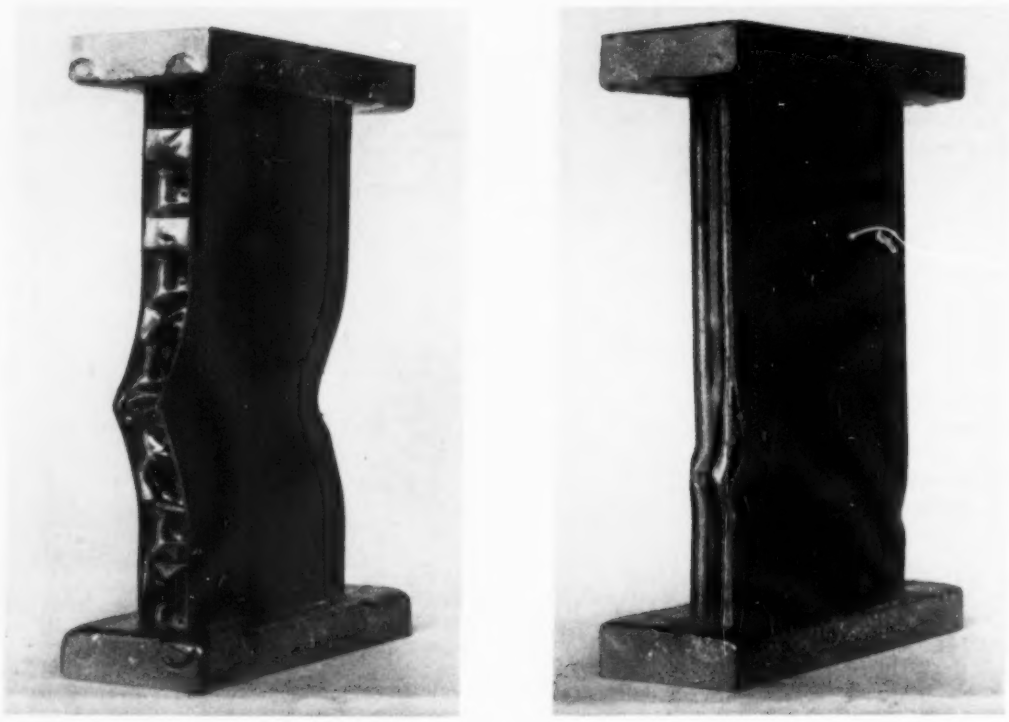
† *The panel stress* is the failing load divided by the cross-sectional area of the faces for the honeycomb panels, and by the cross-sectional area of the faces plus core for the corrugated panels.

TABLE III.—EFFECT OF BRAZING TIME ON JOINTS MADE WITH TERNARY SOLDERS (Bases: Ti–6%Al–4% V; Webs: Ti)

Brazing Time (min.)	Solder: 69% Al–30% Ag–1% Mn		Solder: 69% Al–30% Ag–1% Ni	
	Failing Load (lb./in. run)	Mean Value (lb./in. run)	Failing Load (lb./in. run)	Mean Value (lb./in. run)
10	1145–1590	1444	1376–1570	1482
15	1516–1620	1594	1455–1550	1513
20	1326–1520	1446	1419–1552	1504

TABLE IV.—COMPARISON OF AVERAGE STRENGTHS OF VARIOUS BRAZED JOINTS

Material		Solder: 69% Al–30% Ag–1% Mn		Solder: 69% Al–30% Ag–1% Ni	
Base	Web	No. of Tests	Mean Strength (lb./in. run)	No. of Tests	Mean Strength (lb./in. run)
Ti–6% Al–4% V	Ti–6% Al–4% V	4	1541	4	1453
Ti–6% Al–4% V	Ti	8	1454	8	1580
Ti–5% Al–2½% Sn	Ti	8	1345	8	1495
Ti–6% Al–4% V	Ti–5% Al–2½% Sn	8	1361	8	1343
Ti–5% Al–2½% Sn	Ti–5% Al–2½% Sn	8	1276	8	1411
Ti	Ti	4	1347	4	1404



(a) (a)

Fig. 3.—Failure of sandwich panels in end compression (titanium cores): (a) honeycomb core; (b) corrugated core.

alloy, the nickel solder gives joints of equal or slightly greater strength than the manganese solder.

Sandwich Panels Brazed with Ternary Solders

The results of end-compression tests of sandwich panels made using the ternary solders are shown in Table V. The values obtained for the equivalent and panel stresses are similar to those for the binary alloy, but a marked increase in ductility of the joints was apparent. When failure occurred, the violent rupture observed with the binary solder was absent, and the face plates remained attached to the cores. In the panels with corrugated cores, the brazed areas exhibited a marked tendency to follow the contour of buckling without rupture of the joints, thus indicating that the joint strength is of a similar order to that of the core. In some of the honeycomb panels, tearing of the core and buckling of the face plates occurred without failure of the brazed joints (Fig. 3).

Microscopical Examination of Brazed Joints

A microscopical examination was made of sections through joints between Ti–6% Al–4% V alloy bases and webs of commercially pure titanium and Ti–5% Al–2½% Sn alloy using both of the ternary solders. Photomicrographs of one of these sections are given in Fig. 4 and it is evident that little erosion of the parent metals has occurred, the joints being formed by diffusion of the solders into the parent metals. The zones of diffusion can be seen at the higher magnification. All of the parent metals showed normal microstructures, and the degree of penetration of the solder was approximately the same in all of the joints.

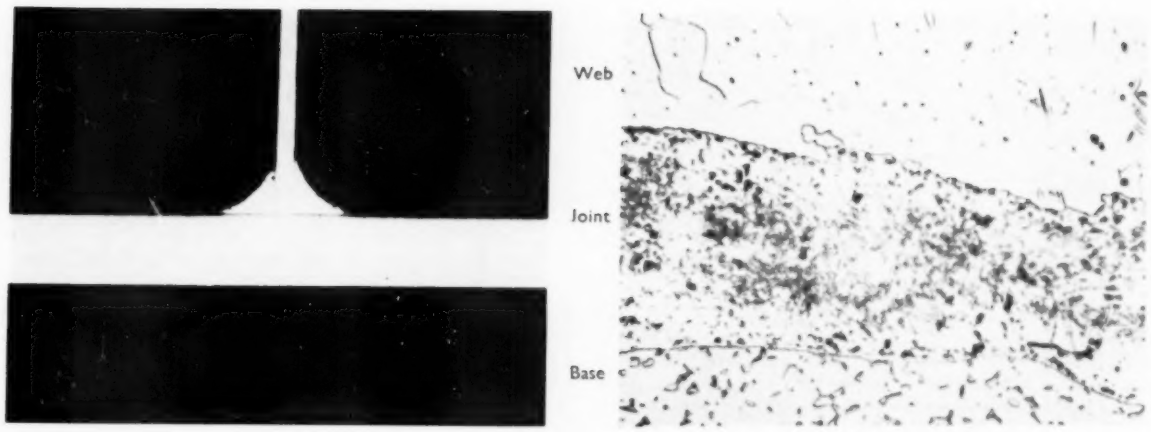
TABLE V.—END-COMPRESSION TESTS OF SANDWICH PANELS BRAZED WITH TERNARY SOLDERS

No. of Panels	Core Type	Solder	Equivalent Stress (tons/sq. in.)	Panel Stress (tons/sq. in.)
4	Corrugated	69% Al-30% Ag-1% Ni	31·5-42·4	27·3-43·6
4	Corrugated	69% Al-30% Ag-1% Mn	34·5-37·8	34·5-37·3
5	Honeycomb	69% Al-30% Ag-1% Ni	44·8-46·3	50·7-57·9
3	Honeycomb	69% Al-30% Ag-1% Mn	45·5-46·0	51·1-55·3

Strength of Brazed Joints at Elevated Temperatures

Tensile specimens were prepared in order to determine the strength of joints at 150°, 300° and 400° C. For each combination of parent metal and solder, four specimens were brazed simultaneously, two being kept at room temperature controls whilst the remainder provided duplicate tests at the elevated temperatures. In view of the fact that some considerable delay was likely to occur between brazing and testing, the room temperature controls were tested coincidently with the hot tests, so that all specimens would be subjected equally to any ageing effects which might occur during storage. The time-cycle for the hot tests was to bring the specimens to temperature over a period of 30 minutes and to maintain them at temperature for a further 30 minutes before testing. The results are given in Fig. 5.

A curious feature of these graphs is that, where the joint consists wholly or in part of commercially pure titanium, the curves are convex towards the origin and are similar to the stress temperature curve for commer-



(a) × 7 (b) × 500

Fig. 4.—Structure of joint between I.C.I. 130 Alloy web and Ti 6% Al 4% V base with 69% Al 30% Ag 1% Ni solder: (a) unetched; (b) etched in 1% HF 12% HNO_3 solution for 6 seconds.

cially pure titanium. For joints made entirely of titanium alloys, the curves are concave, which is the reverse of the stress temperature curves for the alloys. Microscopical examination of a number of joints tested at 150° C and 400° C, showed that at no time did failure occur in the parent metal. There was no evidence of diffusion from the web through the braze metal to the interface with the base-plate, so that it is improbable that the strength temperature properties of the parent metals influence those of the brazed joints.

The two alloys used in these tests both contained aluminium as a major constituent, and it was considered of interest to determine whether similar effects occurred with an alloy not containing aluminium. For this purpose the binary Ti–8% Mn alloy (RCI30A) was chosen, and in order to reduce the number of specimens, joints were made using only the 1% Mn ternary solder. The results of these further tests showed that, for joints made between alloy and alloy, the concave shape of the curve is confirmed. The values for the joints made between titanium and Ti–8% Mn alloy lay on a straight line, and although this did not conform to the convex curves obtained with the aluminium-bearing alloys, it did substantiate the significant difference in strength temperature properties. The reason for the difference is not known.

Influence of Ageing at Elevated Temperatures

The first series of tensile specimens to be tested at elevated temperatures were those at 300° C., and there appeared to be a closer agreement between duplicate results than had previously been obtained at room temperature. It was thought that this might be due to some ageing effect having occurred during the heating cycle, but further ageing tests conducted at 300° C. for periods of up to 24 hours did not yield any conclusive evidence.

Corrosion

For a quantitative assessment of susceptibility to corrosion by sea-water, sixteen panels were made, half having corrugated and half having honeycomb cores. The faces were all of Ti–6% Al–4% V alloy and the cores of commercially pure titanium. Four of each type of panel were brazed with the 1% Mn and four with the 1% Ni ternary solder, thus providing eight pairs of panels. Four of the pairs were exposed to the sea-water spray in an open shed for 3 months, the remainder being kept as controls. At the end of the corrosion test, all the specimens were tested in end-compression to determine the loss of strength as a result of corrosion. The results are given in Table VI, from which it is evident that joints made with the 1% Ni solder are less susceptible to corrosion than those made with the 1% Mn solder. The extent of corrosion is considerably greater on the panels having corrugated cores; this was to be expected since, with these panels, the sea-water spray could be directed along the whole length of the joints, whereas with the honeycomb panels the spray is confined mainly to the outer edges of the panels. A visual examination of the corroded panels before mechanical testing revealed extensive attack where the sea-water had been in contact with the solder, and in all panels the face-plates had started to peel away from the cores. In all specimens corrosion was confined to the soldered joints, there being no evidence of attack on the parent metals.

Sandwich Panels with Titanium Alloy Cores

The ultimate aim of this work was to produce sandwich panels having both core and face-plates of titanium alloy, since a stronger assembly would be obtained than with cores of unalloyed titanium. Since, at present, alloy material less than 0·032 in. thick is not available, it has not been possible to make honeycomb cores in titanium

TABLE VI.—STRENGTH OF SANDWICH PANELS AFTER THREE MONTHS EXPOSURE TO SEA-WATER SPRAY

Core	Solder	Corroded or Uncorroded	Panel Stress (ton/sq. in.)	Mean Value (ton/sq. in.)
Corrugated	69% Al–30% Ag–1% Ni	C	38·7, 39·9	38·8
		U	40·3, 43·6	42·0
	69% Al–30% Ag–1% Mn	C	21·0, 12·5	16·8
		U	34·5, 37·3	35·9
Honeycomb	69% Al–30% Ag–1% Ni	C	32·9, 39·0	36·0
		U	53·0, 57·9	55·4
	69% Al–30% Ag–1% Mn	C	36·9, 26·3	31·6
		U	54·3, 55·3	54·8

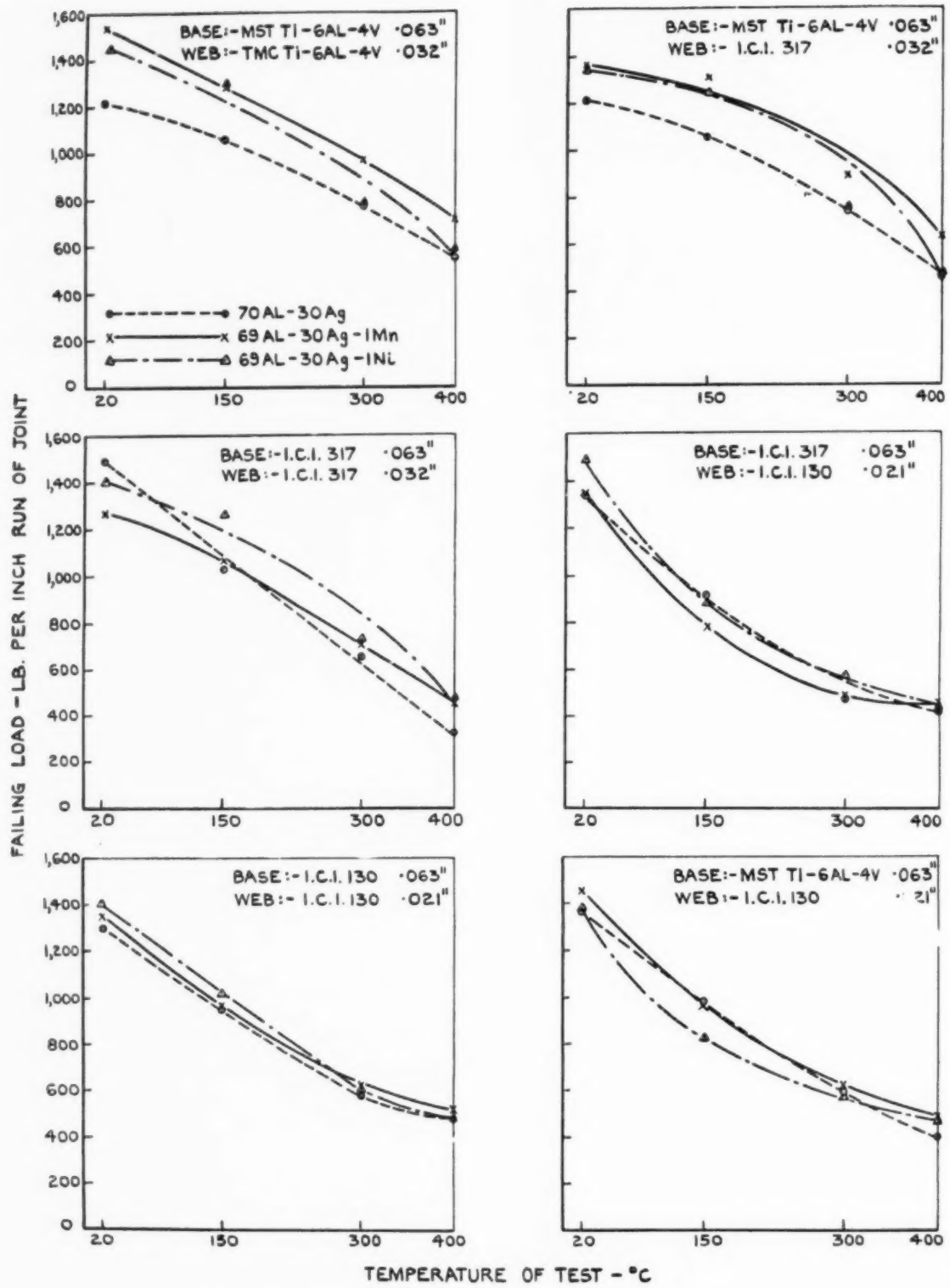


Fig. 5.—Tensile tests at elevated temperatures on brazed 'T'-joints.

alloy, and attention was confined to preparing corrugated cores in 0·032 in. thick Ti–6% Al–4% V alloy.

Unlike pure titanium, this material could not be bent cold through more than 90° round a radius of less than 5T, and it was necessary to resort to hot-forming. For this purpose a two-stage die was used whereby, in the first stage the corrugations were pressed individually, the material being given about 75% of its total deformation. In the second stage the corrugations were fully shaped by one or two presses over the whole of the form. The pitch of the corrugations was 0·9 in. with a vertical height of 0·6 in. and inner radius of curvature 0·140 in. (5T). It was found necessary to heat the material to 950° C. for the first stage and to 1,000° C. for the second in order to eliminate "spring-back". The total heating and reheating time for each core was 15–20 minutes and all heating operations were performed in an atmosphere of argon supplied direct from a cylinder without subsequent purification. After forming, the cores were cut approximately to size on a band saw and pickled in a nitric acid-hydrofluoric acid bath prior to brazing. After brazing, the panels were machined to size (6 × 3 in.) and tested in end-compression.

It will be seen from Table VII that, with one exception, the values for the panel stress all lie within the range 53–56 tons/sq. in. As with the panels having unalloyed cores, collapse of the cores occurred causing dimpling of the face plates prior to rupture of the brazed joints. The panel stress values are approaching the nominal 0·1% proof stress of the "as received" material (60 tons/sq. in.) and there is reason to believe that heating this alloy to 900°–1,000° C. causes some loss of proof stress. To determine whether loss of tensile properties had resulted from the hot-forming, standard tensile test pieces were cut from the undeformed surplus from two of the cores and the tensile properties measured. The results showed the 0·1% proof stress (47 tons/sq. in.) and elongation of the hot-worked material to be appreciably below that of the material as received, and that the failing stress of the panels is close to the proof stress of the latter. The ultimate strength and Young's Modulus were unaffected.

TABLE VII

Solder	Panel Stress (tons/sq. in.)	Equiv. Stress (tons/sq. in.)
69% Al–30% Ag–1% Ni	48·8–55·8	57·2–58·0
69% Al–30% Ag–1% Mn	53·0–56·4	57·6–61·4

Conclusions

(*a*) On a laboratory scale, brazed joints have been made in commercially pure titanium and two of its alloys using aluminium-rich silver alloys as brazing metals. The best results were obtained with either 69% Al–30% Ag–1% Ni or 69% Al–30% Ag–1% Mn.

(*b*) Brazing may be conducted *in vacuo* or in an inert atmosphere such as purified argon.

(*c*) Brazing times of 10–15 minutes are required, the optimum brazing temperature being 670–680° C., but a range of 640°–670° C. is permissible.

(*d*) Tensile strengths at room temperature range from 1,270–1,550 lb. in. run of joint, according to the metals used for the joints.

(*e*) Tensile strengths at 300° and 400° C. are approximately one half and one third, respectively, of the strengths at room temperature. Joints consisting of alloy brazed to alloy retain appreciably greater strength at elevated temperatures than joints between titanium and alloy or titanium and itself.

(*f*) Sandwich panels having corrugated or honeycomb titanium cores give failing stresses of 30–40 and 50–55 tons/sq. in., respectively. Panels having both core and face-plates of Ti–6% Al–4% V alloy give failing stresses of 53–56 tons/sq. in.

(*g*) The brazed joints are reasonably ductile, but suffer appreciable corrosion in sea-water.

Acknowledgments

The author wishes to thank his colleagues, Miss J. A. Gray for experimental work, and Mr. W. Stalker for preparing solders and forging titanium alloy cores.

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3. Lewis W. J., Riepnel, P. S., and Voldrich, C. P., "A Preliminary Report of the Brazing of Titanium to Titanium and Mild and Stainless Steels," *Sheet Metal Ind.*, **32**, Nov., 1955, 846.

Low Cost Refining of Molten Metals

(*continued from page* 204)

lining is in use and gives satisfactory lives. It is rammed around a normal type former and presents no special problems.

The properties obtained on vacuum-refined metal are particularly gratifying, since they come from a furnace which produces the desired effects at a fraction of the cost of an equivalent conventional vacuum melter. With a reasonable degree of utilisation, actual costs are only a few pence per pound of metal treated.

The mains frequency de-gassing and vacuum refining furnace can bring practically all the benefits of vacuum melting into the foundry and melting shop at an extremely low capital and operating cost. It provides a tool which gives close control over gas content, chemical composition and metal temperature. This vacuum treatment method involves relatively small changes in an existing melting shop and, since the equipment normally has fully automatic control gear and ample safety interlocks, it does not demand the extensive training of operators.

Midland Silicones News-Sheet

SOME 35,000 copies of a new "controlled circulation" newsletter have been mailed by Midland Silicones, Ltd., to actual and potential customers of the company. Initially appearing bi-monthly, the *Midland Silicone News* will feature information about the ways in which silicones are used today in almost every industry. The company feels that the new publication will capture the interest of people who do not realise the full extent of the uses of silicones in this country. They have found that all too often people using silicones for specific applications in their own profession have very little knowledge of the ways in which these new chemical compounds are used in other industries—yet this could often be helpful in their own particular fields of interest. Anyone wishing to receive copies of *Midland Silicone News* should write to the head office of the company at 68 Knightsbridge, London, S.W.1.

Antifreeze and Corrosion

THE British Cast Iron Research Association has been engaged for the last two years on a study of the factors influencing the development of excessive rates of corrosion in the waterways of the engines of certain road vehicles using antifreeze solutions produced in accordance with Specification D.T.D. 779 (glycol inhibited with triethanolamine phosphate/sodium mercaptobenzothiazole).*

The evidence available from engine manufacturers' records indicates that the vehicles most liable to severe corrosion are heavy duty diesel engined trucks built during the winter months and filled with the coolant at the factory. Corrosion difficulties usually arise within the first two or three months of service. There is some evidence to suggest that the corrosion is associated with the development of leaks in the cylinder-head gasket.

Analysis of samples of coolant taken from vehicles which have been operating under typical service conditions have indicated that the contents of the inhibitors in the coolant decrease with time, the phosphate and mercaptobenzothiazole being lost at the highest rates. Comparison of the decrease in phosphoric acid and triethanolamine contents and the *p*H of the resultant solution indicates clearly that, at the same time that inhibitor depletion takes place, additional acid is introduced into the coolant. It has been shown that the major portion of this additional acid is formic acid, and there is evidence to show that this arises from the oxidation of the glycol.

Determination of the residual oxygen content in the coolants of engines operating under typical service conditions has suggested that the rate of diffusion of oxygen from the atmosphere to the corroding interface is the controlling factor in the corrosion process, and this suggests that the reason for the apparent correlation between poor gasket sealing and corrosive conditions may be that, on the firing stroke, excess oxygen associated with the combustion gases is forced from the cylinder into the waterways. This oxygen would be at a very high temperature, and might therefore be expected to cause oxidation of the glycol more readily than can be achieved by merely heating the glycol to typical coolant temperatures in the presence of air.

It seems likely that some oxidation of the glycol in an anti-freeze solution, whatever its inhibitor system, will always take place under service conditions. In the case of antifreeze solutions produced in accordance with D.T.D. 779, the rapid corrosion occasionally observed is probably due rather to the fact that the inhibitor system is insufficiently stable than to the fact that in this antifreeze solution glycol is more readily oxidized.

It is thought that the prime reason for the inadequacy of the inhibitors in antifreeze solutions produced in accordance with D.T.D. 779 is the fact that an insufficient concentration is present. The original work on this antifreeze was carried out on solutions of a concentration not less than 30% (i.e. an increase of 50% over that commonly used in road vehicles).

The *p*H value of an antifreeze solution is probably not an adequate criterion of its corrosivity, since a solution of *p*H value as high as 7·5 has been found to be associated with very rapid corrosion, whereas previously it had been assumed that all solutions of *p*H value greater than 6·4 were safe. A better criterion, although one which is much more difficult to determine, is the relationship between the total base and the total acid in the solution. In a solution in which formic acid is being generated at a low rate, the surplus of total base over total acid present in the antifreeze initially will probably be maintained throughout the life of the coolant, but in a solution in which formic acid is being generated rapidly, the surplus of total base over total acid will decrease, and eventually a surplus of acid may be present. It is thought that when a surplus of acid is present the solution can be regarded as potentially dangerous, whatever its *p*H. Fig. 1 gives an indication of the trends which may be encountered in vehicles.

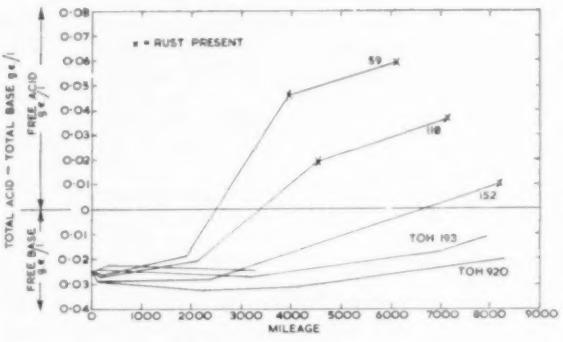


Fig. 1.—Increase in "free acid" in antifreeze solutions with mileage.

I.C.I. Silicon Progress

EARLIER this year I.C.I. announced the bulk production of semi-conductor grades of silicon from their plant—the first in Britain—and it was stated there were plans for expanding the capacity still further to 4,000 lb. The company's faith in the future of silicon has been well justified. The extension has been completed, and the benefit of increased production is being passed on to the consumers by way of price reductions. The demand for silicon in Britain can be met and considerable quantities are being sold abroad, so that extensions have been planned which will more than double capacity in 1960. These modifications will be made relatively simply, as the original plant was designed for ready expansion.

Two distinct types of silicon are required at present by device manufacturers: lump or granular silicon from which single crystals are made; and zone-refined silicon in rod form. Lump silicon (150–500 ohm. cm., p-type) is the form that I.C.I. have been marketing successfully so far. I.C.I. have been actively working on methods of growing silicon rods suitable for zone-refining, and are now installing a new plant which will be producing polycrystalline rods early in 1960. The rods will have diameters up to 1 in. and lengths up to 24 in.: they will have a smooth surface finish and this, together with their very small variations in physical dimensions, will enable them to be used without further processing in zone-refining equipment. It will be possible to obtain high quality single-crystal rods after a few passes in the zone-refiner. The company is continuing active research in close co-operation with device manufacturers in order to keep abreast of new developments.

* *B.C.I.R.A. Journal of Research and Developments*, 1959, **7**, June, 687–691.

Iron and Steel Research

BISRA London Laboratories' Open Days

MAINLY for geographical reasons, it is customary for the British Iron and Steel Research Association to organise its Open Days on a regional basis. The BISRA laboratories are located in London, Swansea, Sheffield and Normanby, near Middlesbrough, and this year it was the turn of the London Group Laboratories to present a visual account of progress in the various investigations in hand. Of the five divisions and three departments into which BISRA is organised, two divisions (Plant Engineering and Energy, and Ironmaking) and the three departments (Physics, Chemistry and Operational Research) have laboratories in London. With the exception of the Ironmaking Division's Blast Furnace Laboratory at Imperial College, all these are housed at Battersea, where most of the laboratory buildings were built before the turn of the century. A programme of rebuilding is in progress, and a new four-storey block of offices and laboratories has recently been completed. The remainder of the site will gradually be rebuilt in accordance with a progressive plan for complete re-development.

At the Sheffield laboratories, three divisions (Steelmaking, Metallurgy and Mechanical Working) are housed; at Swansea, the Sketty Hall premises accommodate the Coatings Laboratories of the Mechanical Working Division; and at Normanby the Ironmaking Division supplements the work carried out in London with pilot-plant work and application trials. The work of the London Group Laboratories thus represents about one-third of the Associations' activities.

The exhibits and demonstrations featured at this year's Open Days represented but a fraction of the projects in hand or recently completed, but in the space available it will only be possible to make brief reference here to a selection from them.

Plant Engineering and Energy Division

Steelworks Cranes

With a view to achieving more economical design of iron and steelworks buildings, BISRA's work on crane gantry girders has been directed towards assessing the dynamic loading effects of the crane wheels they support, the behaviour of the girders under such loading, and the correct method of calculating the stresses and deflections induced. The results of tests at full size and to one-fifth scale have been the subject of BISRA reports and published papers. The impact effects due to crane wheels in motion have been shown to be considerably less than those allowed for in current specifications, but detailed examination of the test results has revealed high local stress concentration in the flange and web immediately under the wheels. Before any reduction in the existing specified impact allowances can be recommended, therefore, a satisfactory method of allowing for these local stresses must be found. Progress is being made towards relating these stresses to the characteristics of the individual girder and to evolving a simple formula capable of being used in conjunction with reduced impact factors. This work should make it possible to design more economical girders and still have an adequate safety margin.

The crane structure itself has also been the subject of study by the Civil Engineering Section, and in view of the importance of fatigue conditions, work is at present in progress to determine the number of times that stress variations occur in the lifetime of a crane. Use is being made of a strain gauge of the mechanical counting type originally designed by the British Shipbuilding Research Association. The instrument on show has been used to record eight stress ranges up to 4 tons/sq. in., and two more pairs of contacts are available to record up to 5 tons/sq. in. The instrument records the number of times each stress—$\frac{1}{2}$, 1, $1\frac{1}{2}$, 2 tons/sq. in. etc.—is exceeded, but the application of a stress of 3 tons/sq. in. will also count as an application of $\frac{1}{2}$, 1, $1\frac{1}{2}$, 2, and $2\frac{1}{2}$ tons/sq. in. This gauge is simple, but its length of 24 in. makes it insensitive to localised strains, and as these may be much higher than the average strains, efforts are being made to use the small voltage signal obtainable from an electric resistance strain gauge to actuate the recording head.

The Tallimarker System

The Tallimarker system demonstrated by the Electrical Engineering Section has been specially designed for conveying and recording information in steelworks. This usually flows in two directions: details of a customer's order come from the mill office and details of progress are transmitted back to the office, where they are used in planning production, making up accounts and compiling reports for managements. At present this information is usually passed on paper chits or by word of mouth—systems which are inefficient, and consequently expensive. The Tallimarker automatic information handling system, on the other hand, is reliable, robust and accurate, and it can reduce clerical work and save time and money.

At key points along the production line of, say, a rolling mill, the operators have push-button desks and display screens, and information can be fed into the system at any of these stages. By pressing a button, an operator can see on his screen relevant information that has been fed in at earlier stages. At the soaking pits, the cast number, ingot number, quality code number and bloom size required are fed into the Tallimarker store, and at the primary mill the Tallimarker is advised whether the ingot is accepted or rejected. At the bloom shears the number of blooms from the ingot is recorded. Further "accept" or "reject" buttons can be operated at the section mill, and a second station at this mill is given information describing the section being rolled. While a section is being cut at the saw, the Tallimarker store holds its complete history, having followed it down the production line from the time the parent ingot left the soaking pit. Each time a length is cut from the section, the details in the store are relayed to the mill office and recorded on a teleprinter, on punched card, or on magnetic tape. The order number and length cut are also recorded, and in addition are displayed to the cooling bank operators to assist in sorting the bars.

Laboratory "mock-up" of the Tallimarker system as it might be installed on a steelworks rolling line. The push-button stations, display screens and central "memory" unit are all shown

"Accept" buttons may be operated by the ingots or blooms themselves, but "reject" buttons must, of necessity, be hand operated. When this happens, the information in the Tallimarker store can be taken out on a punched card, to be fed in again if the rejected material is returned to the production line after reheating.

Hydraulic Gauge Control

The BISRA gaugemeter system of screwdown control for strip mills uses the mill itself as the measuring instrument, the rolls of the mill acting as the rollers of a contact gauge, with compensation for deformation of the mill rolls and housing under load. The limiting factor with screwdown control is the speed with which screw setting adjustments can be made with the normal electric motor drive, and the use of hydraulics is being investigated as a means of speeding up the adjustment and improving the quality of the strip.

The roll load is controlled by varying the force exerted by double-chambered hydraulic capsules inserted one on each side of the mill, between the screws and the top chocks. Each dual capsule contains a large (primary) and small (modulating) chamber. Deformable rubber bonds connecting the two halves of each capsule seal off the chambers from each other and from the atmosphere. The primary chamber is connected to a storage vessel and filled with fluid to form a liquid spring, resisting compression of the capsule and forming an energy store. The primary chamber provides the major part of the roll load, and the modulating chamber provides an additional load which can be controlled. Variations in the roll gap cause relative movements between the two halves of the capsules, and these movements are transmitted mechanically to the spools of the control valves. Compensation for the deformation of the mill components under load is effected by a pressure transducer, which gives a mechanical output proportional to the load and resets the linkage to the control valve.

Screwdown Position Control

Manual control of screwdown drives for rolling mills involves the operation of a multiple controller, each step of which corresponds to a given speed of the screwdown motor. Skill and an ability to concentrate are called for in the operator to enable him to switch off at the right moment to avoid overshooting. The position control system developed by BISRA—where the roll setting is chosen and the screwdown motors started-up and switched-off automatically—offers the dual advantages of greater accuracy and more rapid operation. It may be used in three ways: "follow-up," "drafting" and pre-set. In the first, the screwdown follows-up each movement of the operators lever or wheel; and in the second the initial roll setting is selected by a switch and further small movements take place by pressing drafting buttons. With pre-set control the sequence of rolling is set in advance, and each roll movement is initiated by the operator when required. This method is especially suitable where long rolling pass sequences have to be followed closely.

A potential divider is connected to the screwdown motor and gives a voltage proportional to the position of the rolls. This is electrically compared with a voltage corresponding to the chosen position, and any difference between these two voltages is amplified and used to actuate the screwdown. A number of rolling schedules can be set in advance in electrical form, and to ease the task of the operator in selecting the correct one, the Translator was developed. In this a number of switches are set according to what the operator knows about the ingot to be rolled: size, weight, temperature, finishing size, etc. Each combination of switch settings has the effect of selecting the appropriate rolling programme, and it is only necessary to press a "next pass" button to set the rolls to each successive stage in the programme. More than a hundred programmes can be stored.

Co-ordinated Press and Manipulator

In forging operations the press (or hammer) which deforms the hot metal and the manipulator which manœuvres it during the process are designed independently and controlled by separate means. Difficulty in synchronising and mis-matching of their performance characteristics detract from efficient operation, and the Mechanical Working and Plant Engineering and Energy Divisions, with the help of two hydraulic companies, are engaged in the development of improved types of forging plant to overcome these difficulties.

A combined press and manipulator built in the Sheffield laboratories has a capacity of 200 tons, and is provided with a modern rapid hydraulic control system which speeds up its operation so that each forging cycle can be completed in a second, allowing 1 in. penetration of the

ingot at maximum load. The manipulator was built to keep pace with the press, and is capable of moving a 600 lb. ingot through a distance of 3 in. or an angle of 30° in about one-third of a second; it is also capable of raising or lowering the ingot.

Speed-up of the manipulator movements is achieved by a number of design features. The weight is reduced by having the traversing power unit fixed, with a pinion and toothed rack drive, and the lifting and rotating motions are actuated by a hydraulic cylinder and a compact hydraulic motor, respectively. The hydraulic power unit is also fixed on the floor and not carried by the manipulator.

A servo control system is provided through solenoid-operated hydraulic valves of the simple on/off type, with potentiometers to indicate position, and tacho-generators to measure speed and give the necessary advance warning so that the signalled position can be reached accurately. The control of the press is also arranged in this way. The ultimate aim is automatic co-ordination of both press and manipulator, using a programming circuit which will provide for a complete series of forging passes with either rotary or linear movements of the ingot or a combination of both.

Induction Heating

In spite of the obvious merits of mains frequency induction heating—no loss of heat in waste gases; rapid heating with less scaling and with smaller temperature gradient—it has not been applied in this country to large masses of steel, except in special cases such as the heating of rounds before extrusion. Work on 6 in. round billets has established that induction heating can compete with conventional heating furnaces, on the basis of both fuel cost and fuel quantity, and further work is in progress on the possible applications. This involves the consideration of new handling techniques; new heating schedules to establish maximum heating rates; the economics of changing frequency to match stock size with electrical efficiency; the design of coils to fit different stock shapes; and the fitting of an induction plant into forge shop schedules.

Fluidised Beds

When a stream of air (or other fluid) is passed upward through a bed of sand (or other powder) so that the particles of sand become suspended in the airstream, the resulting suspension resembles a liquid. Such a system is called a fluidised system, and the sand-air suspension is known as a fluidised bed and behaves as a fluid. The thermal conductivity of a fluidised bed is greater (by up to one hundred times) than that of the fluidising gas alone so that the heat transfer rate between the walls is also greater. There is no thermal or concentration gradient across the bed, and the pressure drop across it depends on its height and not on the size of the particles.

In the first exhibit, the liquid-like properties were demonstrated by the floating on its surface of a toy duck and the sinking through it of a piece of iron. A bed of this type has been used, surrounded by a water jacket, for mould cooling, and it is hoped that the technique will enable moulds to be cooled rapidly, but uniformly, without the marked reduction in mould life attendant on water quenching. The second exhibit was a hot-bed in which the maximum temperature reached has been about 1,400° C. The use of such beds is being investigated for ingot heating and hot storage. Established uses of fluidised beds in the iron and steel industry include ore roasting and the desulphurisation of coke oven gas.

Temperature Measurement

The work of the Energy Section in analysing furnace performance and investigating problems of heat transfer necessitates obtaining accurate temperature measurements over a large range, and special instruments have been developed for particular uses. In the pyrometer devised to eliminate errors in measuring the surface temperature of an ingot during heating in a forge furnace, a highly reflecting hollow hemisphere is placed in contact with the surface and the brightness of the black body produced is measured by the disappearing filament technique. Other instruments include a suction pyrometer designed to measure the temperature of the corrosive dust-laden waste gases of an open hearth furnace; a twin beam total radiation pyrometer for the measurement of temperature and emissivity of flames; and a radiation flux meter whose purpose is the measurement of heat transfer rather than temperature.

Ironmaking Division

Experimental Technique for Studying the Blast Furnace Process

The solution of many problems in blast furnace practice would be facilitated by the availability of satisfactory experimental techniques for: (*a*) detecting restrictions on productivity and fuel economy in the blast furnace process; (*b*) evaluating the effects of changes in blast furnace operation and design on productivity and fuel economy; and (*c*) evaluating the quality of materials for the furnace. Hopes are high that a new experimental technique will fulfil these requirements, and tests on a small scale have been encouraging.

If one considers an ideal furnace, in which a perfectly distributed charge descends evenly, steadily and without friction against the walls counter-current to perfectly distributed gas, the conditions (chemical composition, temperature, pressure, flow rate, etc.) will remain constant at any given level in such a furnace. If a horizontal slice of the charge be imagined as descending through its own height, the conditions which had prevailed at the bottom will now hold at the top of the slice; in other words, the conditions at the top of the horizontal slice will lag behind those at the bottom by the time the slice takes to descend its own height. The second basic principle of the method is that the difference between conditions at any two levels is due to the interaction between the gas and the charge between those levels. This enables the difference in conditions between levels to be determined by experiments outside the furnace.

The apparatus consists essentially of a container large enough to take a representative portion of the furnace charge, which can be supplied with gas of any temperature, composition and pressure at any flow rate, all these being measurable at the inlet (bottom) and outlet (top) of the container. The charge is fed into the container (through which gas is passing at the same rate, temperature, etc., as at the stockline) so that it builds up there at the same rate as it descends in the furnace. The gas entering the container is continuously adjusted at the inlet so that the conditions at the outlet remain, at this stage of the experiment, those of the furnace stockline. Then,

because the actual furnace charge and conditions have been used, the inlet conditions when the charge height in the container is any given value, must be those at the same distance below the stockline in the furnace. When a suitable charge height has been built up, no more is added and the inlet conditions are continuously adjusted in such a way that the outlet conditions are no longer constant, but lag behind them by the time the particular portion of the charge would take to descend its own height in the furnace. In this way the portion of the charge taken is, in effect, followed down the furnace, time being substituted for motion.

This technique will enable various operating conditions to be reproduced outside the blast furnace and studied far more effectively than is practicable on a working furnace, and far more cheaply.

Sinter

Laboratory work using solid fuels of widely different reactivities has confirmed that the combustion of carbon in a sinter bed proceeds completely to carbon dioxide in spite of the excess oxygen present, and has emphasised the need to take reactivity into account when assessing the suitability of a fuel for use in sintering. Further experiments have showed that the maintenance of two flame-fronts in the bed with a rich-ore mix can only result in strong sinter if the oxygen in the sintering atmosphere is raised to 30%. The use of preheated air and three layers has made possible the production of good sinter with 20% less air. In the case of a home-ore mix, preheating to 70° C resulted in a higher permeability at all stages of sintering and thus a shorter sintering time.

The relationship between strength of sinter and the temperatures recorded in the bed during sintering has been studied, and the results suggest that the strength of the sinter is controlled by different factors according to the quantity and composition of the slag-forming constituents present in the mix.

In a study of the chemical compounds likely to be formed during the manufacture of self-fluxing sinter, briquettes of finely ground mixtures of iron oxide with added slag-forming oxides have been heated in air to temperatures from 1,150°–1,400° C. and analysed by X-ray diffraction photography. Any calcium ferrite formed reacts at higher temperature to give ferric oxide and calcium silicate. On the other hand, magnesium ferrite does not decompose in this temperature range.

With lean-ore mixtures the use of preheated air for about one-third of the total sintering time after ignition can result in a saving of some 15% in the coke breeze required, the total thermal input being reduced. In rich-ore sintering practice, savings in coke are in general less than with home ores and depend on the ore mix used. In extreme cases, there may be no saving of coke ; in no case of rich-ore sintering has the heat required been reduced. The effects of applying hot air before ignition in order to dry out and preheat the bed have also been investigated. The results show a slight thermal advantage over the corresponding treatment after ignition, but the gain is not great enough to justify allocating part of the strand for simple pre-drying of the sinter mix.

(*to be continued*)

Molybdenum in Sierra Leone

MOLYBDENUM has now been found in the Northern Province of Sierra Leone, says a Barclays Bank D.C.O. message from Freetown. The discovery was made after two slanting holes had been bored to probe the rocks underlying the patch at vertical depths up to 450 ft. Cores of rock taken from the holes contain enough molybdenum and lead minerals for experts to advise further drilling. Faint indications of molybdenum are fairly widespread around Lake Sonfon, says the Bank report, and another team will be flying out to Sierra Leone from the Royal School of Mines next year to test streams and river water in about 150 square miles of country for molybdenum minerals. During the past twenty years scattered small occurrences of molybdenum minerals have been reported from time to time, but it was not until 1951 that larger quantities were found.

Non-Ferrous Foundry Health Risks

THE second report of the Joint Standing Committee on Safety, Health and Welfare Conditions in Non-Ferrous Foundries, published by the Ministry of Labour recently* is devoted to known and potential health hazards in non-ferrous founding. The Committee is of the opinion that the report on known risks prepared by its Technical Sub-Committee, to whom the question of health risks was remitted, "warrants the close attention of the industry," which it is urged should act on it as soon as possible. Attention is drawn to a medical appendix covering both known and potential risks, and the Committee comment that a "good knowledge of this part would result in the avoidance of many risks which might be introduced inadvertently with new processes." The Committee agrees with its Sub-Committee in the view that medical supervision is desirable wherever it can be arranged, and that a general medical survey of the industry would help greatly by establishing the type and magnitude of the health risks involved.

* H.M. Stationery Office, price 1s. 9d. net.

Foundry Equipment for Germany

BAKER PERKINS, LTD., Bedewell Division, Hebburn-on-Tyne, have received a contract for the supply of foundry equipment to the well-known German founders E.G.O. Elektro-Gerate, Blanc u. Fischer, Oberderingen. The British firm will supply a completely automatic moulding line to work in conjunction with two 24 × 24 in. Taccone diaphragm moulding machines. The plant will be capable of 150 moulds per hour and will include indexing mechanisms, roll-over and lowering devices, a closing device, automatic box separation and powered conveyor lines.

British Oxygen Head Office

THE British Oxygen Co., Ltd., have completed arrangements to lease a large modern office building near Hammersmith for their head office. Construction will start almost immediately and it is expected that the move from the present premises in St. James's will take place in two or three years' time.

Aluminium in the 'Oriana'

Thousand Ton Welded Superstructure

The all-welded aluminium deckhouse structure in course of construction at Barrow.

WHEN the *Oriana* goes into operation in the new joint Australian and Pacific service developed by the Orient Line and P. & O., she will be noteworthy not only as one of the most advanced passenger-carrying ships in the world, but also for the scale on which aluminium has been used in her construction. By adopting welded aluminium fabrication for the whole of the vessel's superstructure above the strength deck, enough weight has been saved to allow the passenger accommodation to be increased to the extent of a complete deck, without reducing stability. In the eight tiers of deckhouses, three of which extend the full 450-ft. length of the superstructure, 1,000 tons of aluminium plate and sections were used, about 870 tons being supplied by Northern Aluminium Co., Ltd.

Welding

Planning for the use of aluminium on this scale necessarily began several years ago, and preliminary investigation to settle such questions as the best welding procedures and techniques to be adopted was undertaken in 1955. In that year a large experimental deck panel was constructed in co-operation between the shipbuilders, Vickers-Armstrongs (Shipbuilders), Ltd., and Northern Aluminium Co., Ltd. The general method of construction used for the panel was to join the plates by butt welds laid on to permanent backing bars, wherever possible. Although at that time experience of welded aluminium deckhouse construction was not so extensive as it is now, this system had already been successfully used for other ships, and it was eventually adopted for the *Oriana*.

The welding itself was carried out using consumable-electrode argon-shielded-arc equipment. The widespread use of welding as a means of fabricating aluminium structures is comparatively recent, but the modern fluxless processes of this type are now thoroughly established, and have brought the same ease of construction in aluminium that the shipbuilder is accustomed to find with steel.

Among the items of aluminium welding equipment used by Vickers-Armstrongs for work in the *Oriana* was a Quasi-Arc gantry type of machine (capable of Sigma welding of aluminium and Fusarc welding of steel) for fillet welding; Quasi-Arc tractor-mounted equipment for butt welding, both in the shop and aboard the ship; and Quasi-Arc/B.O.C. Lynx sets for manually-operated assembly welding.

Construction

Prefabrication was adopted as an essential technique in the building of the aluminium superstructure. Deck panels with a typical size of 25 ft. sq. and weighing about 2 tons were built up as units, and several larger structures, such as internal stairways, were also fabricated and lifted aboard complete.

As with a steel ship's structure, it is an advantage to use aluminium plates of the largest available size. Until recently, designers have tended to be hampered by the fact that at present there is no hot mill at work in this country capable of supplying aluminium plates in widths greater than about 7 ft. To overcome this problem until their new wide rolling mill comes into operation next year, Northern Aluminium have supplied for the *Oriana* and other applications Noral Welded Plate. This is aluminium alloy plate up to 11 ft. in width, produced by the automatic welding together of two plates edge-to-edge in a special jig. The weld bead on the resulting composite plate can if desired be ground flush, on both sides if necessary, making the finished plate comparable in appearance with ordinary rolled material. The quality of the welding, moreover, is such that for most purposes not involving forming, the presence of the weld need hardly be considered.

To ensure flatness, many of the pieces of Noral Welded Plate supplied for the *Oriana* were stretched after welding, using a 4,000-ton stretcher recently installed at Northern Aluminium's Rogerstone works. This huge machine, the only one of its kind in service in Europe, made a significant contribution to the success of the aluminium plate supply. Similarly important was a 60-ft. plate saw, in use at the same works, capable of cutting plates to a hitherto-unattainable degree of accuracy and with guaranteed edge straightness. By the use of this equipment it was possible to supply the aluminium plates cut to the exact required widths, thus

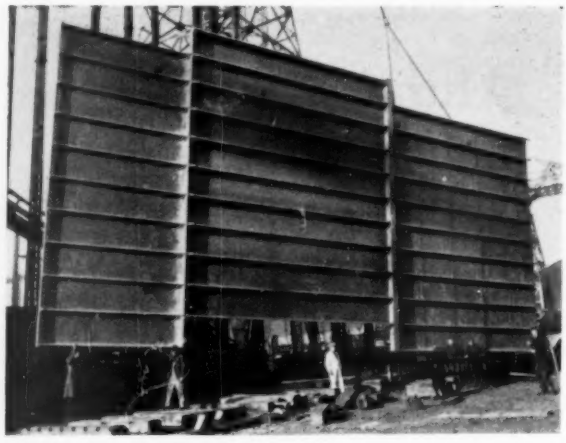
reducing the amount of shipyard plate trimming work to the minimum.

The general thickness of the plates supplied was from 0·20–0·40 in., with a small number of thicker plates. All were of aluminium alloy to the B.S. NP5/6 specification, meeting the requirements of Lloyd's Register for welded ship structures.

As far as possible, extruded aluminium alloy sections were used in the *Oriana's* superstructure for girders and other members, as well as for the usual deck beams and stiffeners, rather than members fabricated from plate. This has meant that the proportion of extruded sections in the structure is higher than is usually the case in work of this kind.

The sections throughout were of aluminium alloy to the B.S. NE6 specification, again meeting the requirements of Lloyd's Register. Some unusually large extrusions were included, up to 16 in. in overall dimension and with a weight per ft. of over 15 lb. For beams and stiffeners incorporated in the prefabricated deckhouse panels, a special series of sections with a tapered bulb was used, as an alternative to the better-known square-bulbed British Standard aluminium shipbuilding sections.

The building of a ship with this 1,000-ton aluminium superstructure—incidentally by far the largest welded aluminium structure every built—not only indicates the progressive outlook of those responsible for its design, but at the same time clearly demonstrates the great progress that has been made in the development of sound techniques for the employment of welded aluminium in shipbuilding. Less than ten years ago a shipyard application of aluminium on the scale of that in the *Oriana* would hardly have been predictable; the advances made, particularly in the welding of aluminium, have undoubtedly surpassed all the expectations of a few years ago.



Preliminary investigation work to assess the best welding procedures and techniques included the construction of this experimental deck panel, built in 1955 as a result of co-operation between Vickers-Armstrongs (Shipbuilders), Ltd., and Northern Aluminium Co., Ltd.

All-Electric Melting Shop Planned

Steel, Peech and Tozer, of Rotherham—branch of The United Steel Cos., Ltd.,—are to spend £10m. on the replacement of all their existing open hearth steel melting furnaces by six electric arc furnaces of 110 tons capacity each. When completed in about five years' time, this will be the largest electric steelmaking plant in the world, with an annual capacity of 1,350,000 ingot tons of steel. It is also the largest single development scheme in the branch's eighty-year history.

At the present time, there are twenty-one open hearth furnaces at Steel, Peech and Tozer—fourteen in the Templeborough melting shop and seven in the Rotherham melting shop—producing approximately one million ingot tons of steel per year. The existing Templeborough melting shop is to be adapted to house the new electric furnaces, and this will mean the disappearance of a well-known local landmark with the demolition of the fourteen 120 ft. high chimneys for waste gases, although the present size and shape of the building will remain substantially as at present. The Rotherham melting shop will be demolished entirely.

Initially, four electric furnaces of the swing-roof type will be installed, and it is planned to have the first of these in commission by January, 1963. In order to make a start on the plant, however, the first two open hearth furnaces at the Sheffield end of the Templeborough melting shop will close down in two years' time, to be followed by others as the scheme progresses. Arrangements have already been made to maintain the existing level of steel production during the changeover by fitting double oil burners to some of the furnaces and converting others to "all-basic" refractory linings. No order has yet been placed for the supply of the electric furnaces, but almost everything apart from the electrical equipment is to be manufactured by Distington Engineering Co., Ltd., a United Steel subsidiary.

Negotiations with the electricity supply authority for the supply of power have not yet been concluded, but the power requirements of the new plant will be 750 million units per year—six times the amount used by the whole works at present, and over one-third of the existing industrial load in the Sheffield area. A new electrical sub-station is to be built near to the company's Brinsworth mill which will take power at 275,000 volts from the super-grid. This sub-station will house the Yorkshire Electricity Board's transformers and switchgear and will permit a metered supply of power at 33,000 volts to be conveyed by cable to the Templeborough melting shop, where it will be transformed down to a working voltage of 565/200 for furnace consumption.

The changeover from the open hearth to the electric arc process will entail the provision of new steam generating equipment, as all the existing waste heat boilers will become redundant. Special attention will be given to fume extraction equipment with the aim of minimising the dust nuisance. A new ingot mould preparation building is to be laid down adjacent to the melting shop and this will be twice the size of the existing mould preparation building, which it will replace.

The head office of the Wakefield Group has moved to Castrol House, Marylebone Road, London, N.W.1. (Telephone: Hunter 4455).

NEWS AND ANNOUNCEMENTS

New Vacuum Science Committee

FOLLOWING The Institute of Physics' London Conference on high vacua held in April last, various suggestions were made for arranging regular British meetings on vacuum science and technology, and for British participation in international conferences in this field. As a result of informal discussions between societies and institutes in this country a Joint British Committee for Vacuum Science and Technology has now been formed. The committee consists of representatives from each of the following bodies :—

The Institute of Biology
The Institution of Chemical Engineers
The Royal Institute of Chemistry
The Institution of Electrical Engineers
The Iron and Steel Institute
The Institution of Mechanical Engineers
The Institute of Metals
The Institute of Petroleum
The Physical Society
The Institute of Physics

Its objects are :—

(*a*) to co-ordinate and help to initiate meetings in the whole field of vacuum science and technology arranged by constituent bodies, and

(*b*) to act in the collective interest of the constituent bodies by maintaining liaison with the International Organisation for Vacuum Science and Technology and with national vacuum societies, and otherwise.

The Institute of Physics has agreed to provide the secretariat for the joint committee ; communications should be addressed to the secretary of the joint British committee at its headquarters, 47 Belgrave Square, S.W.1.

Wellman-Vöest L-D Agreement

A CONTRACT between Vöest A.G. and The Wellman Smith Owen Engineering Corporation, Ltd., has been signed in Linz by Herr Hitzinger and Sir Peter Roberts, Bt., for exclusive co-operation in the U.K. for the design supply and erection of plant and equipment for the L-D steelmaking process, which has proved extremely successful on the Continent. A further and more detailed announcement will be made later.

A.E.I. Reorganisation

ON January 1st, 1960, the British Thomson-Houston Co., Ltd., Metropolitan-Vickers Electrical Co., Ltd., and Siemens Edison Swan, Ltd., change their names to Associated Electrical Industries (Rugby), Ltd., Associated Electrical Industries (Manchester), Ltd., and Associated Electrical Industries (Woolwich), Ltd., respectively.

At the same time five new product divisions of Associated Electrical Industries are announced. The Cable and Construction Divisions combine the interests of the S.E.S. Cables Division with those of W. T. Henley's Telegraph Works Co., Ltd., and Liverpool Electric Cables, Ltd. The Telecommunications Division has hitherto traded as a product division of Siemens Edison Swan, Ltd. These three divisions, together with the Radio and Electronic Components Division, will be managed by Associated Electrical Industries (Woolwich), Ltd. (formerly S.E.S.). The Instrumentation Division combines the interests of Sunvic Controls, Ltd., with the instrument and meter, X-ray, and scientific apparatus departments of Metropolitan-Vickers and will be managed by Associated Electrical Industries (Manchester), Ltd. (formerly M-V.).

So from January 1st, 1960, the total of A.E.I. product divisions to be established becomes twelve, and the famous companies whose names are to change will cease to trade, but will manage a number of A.E.I. product divisions. Their employees will be transferred to A.E.I. employment. Birlec, Ltd., the heat treatment and furnace manufacturing company, will be renamed A.E.I.-Birlec, Ltd., and will be managed by Associated Electrical Industries (Rugby), Ltd. (formerly B.T.H.). The name of its associate company, Birlec-Efco (Melting), Ltd., will not be changed.

A.E.I. is thus established as a trading company in the fullest sense—a relatively new name in the world of commerce but with the history and strength of famous companies behind it—and Britain's largest electrical manufacturer.

B.S.C.R.A. Council Elections

DR. R. HUNTER, deputy managing director of the Clyde Alloy Steel Co., Ltd., and a leading authority on alloy steels and steel castings, was recently elected chairman of the council of the British Steel Castings Research Association, having lately been vice-chairman. MR. W. S. SCOTT, a director of Darlington Forge, Ltd., has succeeded Dr. Hunter as vice-chairman of the council.

The retirement of MR. C. H. KAIN, managing director of Lake & Elliot, Ltd., and the National Steel Foundry (1914), Ltd., who was chairman of the council for over five years, was marked by the presentation, by the Association's staff, of a stainless steel gavel, designed and made at the Research Station. This gavel comprises a scale model of a handshank, the anvil being formed of an etched section of a clover leaf test piece—an acknowledgment of Mr. Kain's invention of this design of test piece. An eloquent tribute to the work of the retiring chairman was paid by DR. A. H. SULLY, director of research, B.S.C.R.A., who said that the erection of the Research Station and the expansion now in progress were a lasting testimony to Mr. Kain's chairmanship.

The Computer in Production

THE INSTITUTION OF MECHANICAL ENGINEERS is arranging an informal discussion on "The Computer in Production." The purpose of the meeting is twofold :—

(1) to introduce chartered mechanical engineers and managers to the latest techniques involving the application of computers in production ; and

(2) to provide a forum for managers, engineers and users to present their views to manufacturers of computers.

The meeting will be held on Monday, March 21st, and

Tuesday, March 22nd, 1960, and will commence at 10 a.m. each day, continuing until 8 p.m. on the first day and about 4 p.m. on the second day. Several distinguished engineers and users of computers will give talks on the subject, and the discussion will be informal, and will not be reported. Visits to computer centres and to installations where computers are being used on production problems will be arranged.

This meeting will be open to members and visitors, and is aimed primarily at management level; but the subject treatment is also likely to be of interest to specialists in computers. Mechanical engineers are employed in a very wide range of industries, and there is every indication that a forum is needed for a discussion on computers in "Production" in its widest sense. Those who wish to attend the meeting should apply to The Secretary, The Institution of Mechanical Engineers, 1 Birdcage Walk, Westminster, London, S.W.1.

Metallographic Techniques Course

A COURSE of twelve meetings (seven lectures and five practical periods) on modern metallographic techniques will be held on successive Wednesday afternoons, commencing January 13th, 1960, at the Northampton College of Advanced Technology, St. John Street, London, E.C.1. The syllabus covers recent developments in polishing and etching techniques; thin film preparation; special methods of illumination in microscopy; micro-hardness testing; electron microscopy; microradiography; colour photomicrography; T.V. microscopy; and high temperature metallography. The fee for the course is two guineas and information concerning enrolment can be obtained from the College.

U.S. Tour

DR. A. H. SULLY, Director of the British Steel Castings Research Association, Sheffield, presented a paper on "The Control of Dust and Noise in Steel Foundries" to the Technical and Operating Conference of the Steel Founders' Society of America, which took place in Cleveland, Ohio, from 9th–11th November. Dr. Sully's lecture was illustrated by a film, entitled "Let's Clear the Air", produced by, and obtainable on loan from, the B.S.C.R.A. The invitation extended to Dr. Sully is a tribute to the pioneering work in the field of industrial health carried out by British steelfounders through their Research Association. In the course of his tour, Dr. Sully visited a number of steel foundries and research centres in the U.S.A. and in Canada.

Hilger & Watts—I.R.D. Merger

HILGER & WATTS, LTD., have acquired the whole of the issued share capital of The Infra-Red Development Co., Ltd., Welwyn Garden City, which was founded in 1946 and specialises in the analysis of gases by non-dispersive infra-red techniques, primarily for industrial purposes. From the start, the firm has been under the technical control and management of Mr. W. B. Bartley, who will remain managing director. The pooling of knowledge and experience will be of great value to the two companies, and the technical, financial, and manufacturing resources of the Hilger & Watts organisation will expedite further developments in gas analysis and their application in the expanding field of process control. In that field, dispersive infra-red techniques (in the use of which Hilger & Watts have had long experience) are also likely to become more important. The Infra-Red Development Co., Ltd., will continue to operate at its works and offices in Welwyn Garden City and will continue to develop and manufacture the gas analysers on which it has built its reputation.

Dow British Subsidiary

THE British subsidiary of The Dow Chemical Co. of Midland, Michigan, U.S.A.—Dobeckmun Britain, Ltd.—has changed its name to Dow Chemical Co. (U.K.), Ltd., and broadened the scope of its activities. The company will continue to handle the manufacture and sale of Lurex, a non-tarnishing metallic yarn, produced in Windsor, Berkshire. In addition, it will take over direct sales of Dow-produced packaging films such as Tryeite (Dow polystyrene film), Saran Wrap (Dow vinyl-vinylidene chloride copolymer film), and polyethylene film. It will also supervise the sales of Dow U.S. and Canadian-produced industrial chemicals, plastics and magnesium in the U.K. In the latter line of products, the company will continue to use the services of appointed distributors. Address of the company is Dow Chemical Company (U.K.), Ltd., 48 Charles Street, London, W.1.

Disposal of Tin Stockpile

THE Board of Trade gave notice on June 26th, 1959, that they intended to make available for sale the remainder of their stocks of tin, amounting to about 2,500 tons. The International Tin Agreement requires six months public notice of disposal of non-commercial stocks. The Board of Trade now announce that arrangements have been completed, subject to the formal agreement of the International Tin Council at their next meeting in December, for the sales to take place through the Buffer Stock Manager of the Tin Council, and for the tin to be available to him from December 28th, 1959. Sales of the second 2,500 tons will be made on the same basis as that of earlier sales.

Pipes and Pipelines Exhibition

RECENT technical progress and growing world-wide interest in pipes, pipelines and their ancillary equipment is reflected in the decision to hold the First International Pipes and Pipelines Exhibition. Sponsored by the journal *Pipes and Pipelines*, it will be held at Earls Court, London from 30th May to 2nd June, 1960, and will provide an opportunity for executives, managements and buyers of pipe-using firms to see what the industry has to offer, and to examine at leisure the latest developments, materials, techniques and applications. Further details are available from the organisers: Scientific Surveys, Ltd., 97 Old Brompton Road, London, S.W.7.

Magnesium Price Reduction

ALCAN (U.K.), LTD., have announced a reduction in price of magnesium ingot. Metal of 99·8% minimum purity is delivered customer's works for 2s./lb., a reduction of 3d./lb. on the price current since the beginning of the year.

Stelvetite, the plastic coated steel sheet, was featured by the manufacturers, John Summers & Sons, Ltd., on their stand at the Institute of Sheet Metal Engineers Exhibition last month. The picture shows the stand on which were arranged typical items incorporating Stelvetite, including a Triumph TR3 sports car facia panel, a Scales-Spin dryer, a Santon water heater, clocks by Smiths and A. S. Brown, a Rabone measuring tape, a fireside log-box by Herman Smith Mastercraft, an Arthur Shaw companion set, and a miniature sample of Quiltile acoustic tile.

B.N.F.M.R.A. Chairman

Mr. F. C. Braby, M.C., D.L., (chairman and managing director of Fredk. Braby & Co., Ltd) has been elected chairman of the council of The British Non-Ferrous Metals Research Association to succeed Dr. Maurice Cook, C.B.E. who retires from office on the 31st December next. A Past President of the Engineering and Allied Employers' National Federation and of the London and District Engineering and Allied Employers' Association, Mr. Braby has also contributed much to the managerial efficiency and technical advancement of the galvanizing industry through his service as chairman of the Hot Dip Galvanizers' and the Galvanized Tank Manufacturers' Associations. Mr. Braby has played an active part in the affairs of the Research Association since 1929, and has been a member of council since 1935: in 1950 he was appointed a vice-chairman and hon. treasurer of the Association.

Electric Smelting Pilot Plant

Mr. C. J. Beavis has been appointed by Birlec-Efco (Melting), Ltd., to take charge of all Birlefco's smelting activities. Formerly works engineer for The Distillers Co., Ltd., at their factory near Port Talbot in South Wales, Mr. Beavis was responsible for all engineering construction and maintenance required for the production of calcium carbide. A pilot plant is being commissioned at Aldridge, and Birlefco will be the only company in Britain to operate a contract service for electric smelting. The plant is intended to test the amenability of customers' raw materials to electrothermal reduction, and to determine the most suitable smelting technique and the nature of the products obtained.

Tonnage Oxygen Plant

Appleby-Frodingham Steel Company, a branch of The United Steel Cos., Ltd., are installing a tonnage oxygen plant at their Scunthorpe works at an estimated cost of £750,000. Site work has already begun and the plant is expected to be in commission by the late summer of 1960. Intended for the continuous production of gaseous oxygen for metallurgical and combustion purposes, the new plant is being designed to produce 100 tons of gaseous oxygen per day of 99·5% purity at 600 lb./sq. in. gauge and ambient temperature. Alternatively, 90 tons of gaseous oxygen and 10 tons of liquid oxygen per day can be produced, so that the latter can be stored and evaporated to cover planned stoppages of the main plant.

Principal contractors for the tonnage oxygen plant are Air Products (Great Britain), Ltd., the British subsidiary of Air Products Incorporated, of Allentown, Pennsylvania, U.S.A. All the structural steelwork is being fabricated by United Steel Structural Co., Ltd., a United Steel subsidiary, and the complete installation will be carried out by Woodall-Duckham, Ltd.

Personal News

Mr. C. Fox, partner in Daniel C. Griffith & Co., assayers to the Bank of England, retired from the firm at the end of September after more than fifty-two years service. Mr. A. R. Griffith, a great-grandson of the founder, has joined the directorate.

The Pressed Steel Co., Ltd., announces that Mr. R. N. Davies, works director at Cowley and chairman of the motor car body divisional board has been appointed a local director of the company. Mr. Davies joined Pressed Steel in 1926, shortly after its inception, as a foreman on the assembly lines, becoming a superintendent in the early 1930's. He was appointed to works management in February, 1952, and to the chairmanship of the motor body divisional board in December, 1957.

Mr. B. Williams has been appointed resident technical sales representative at the Bristol office of Crofts (Engineers), Ltd., in place of Mr. Dawson, who has left the company's service.

Mr. P. A. Charlesworth, a director of Electropol Processing, Ltd., and managing director of the patent holding company for the process, Electropol, Ltd., Poynton, Cheshire, recently made a tour of the U.S., where his business was concerned with technical liaison,

exchange of information, reciprocal sales promotion, and the visiting of main research organisations and commercial interests in the stainless steel and light alloy fields.

DR. R. A. MOTT has been appointed consulant to The British Coke Research Association on relinquishing his position as superintendent of the chemical and physical laboratories of the Association's Coke Research Centre, Chesterfield.

MR. H. D. BOYD has retired from the board of Edgar Allen & Co., Ltd. Joining the company in 1906, Mr. Boyd was elected a director in 1933: he continued in an executive capacity until 1956, and retained his seat on the board until September of this year.

THE Steel Company of Wales, Ltd., has announced that following the completion of the company's third development plan, and in view of the retirement of CAPTAIN H. LEIGHTON DAVIES in June, 1960, MR. R. W. EVANS, THE HON. R. H. M. KINDERSLEY, and MR. I. S. SCOTT-MAXWELL have been appointed to the board. Captain Leighton Davies will continue as a consultant to the company following his retirement. Mr. Scott-Maxwell is appointed general manager designate to the tinplate division, to succeed Captain Leighton Davies in June, 1960, and MR. SHIRLEY GAZARD, assistant general manager—tinplate division, is appointed deputy general manager to the tinplate division from the same date. MR. R. W. EVANS, at present assistant general manager (operations), steel division, is appointed deputy general manager, steel division. The Hon. R. H. M. Kindersley joins the board as a non-executive director.

As a result of appointments announced by English Steel Forge and Engineering Corporation, Ltd., MR. R. W. BROCKLEHURST, director, becomes general superintendent; MR. E. LEVESLEY, manager, becomes superintendent of the forge machine shops; and MR. R. J. H. HUNT, manager, becomes superintendent of the heavy forge.

MR. C. W. V. DAVIS, a director of Research and Control Instruments, Ltd., will head the newly-formed industrial products division of that company, which will in future distribute Philips arc welding supplies, filtration equipment and high frequency generators.

A SOUTH WALES area sales office has been established by Birlec, Ltd., in Cardiff, and MR. W. H. STATHAM, who is appointed area representative for South Wales, has his headquarters at Associated Electrical Industries, Ltd., Mervyn House, Frederick Street, Cardiff. (Telephone: Cardiff 28511). Mr. Statham joined Birlec in 1946 and, prior to his new appointment, was a sales engineer in the furnace division of the company.

WICKMAN, LTD., announce the appointment of MR. R. J. DIXON as director and general manager of their machine tool manufacturing company.

MR. N. R. CRUMP, president of the Canadian Pacific Railway Co., has been elected a director of The International Nickel Co. of Canada, Ltd.

MR. H. SLACK has been appointed to the board of Kelvin & Hughes (Industrial), Ltd., as sales director. Mr. Slack, who joined the company in 1949, was formerly technical sales manager.

MR. P. BEYNON has been appointed works manager, operational research and work study, at Steel, Peech and Tozer, a branch of The United Steel Cos., Ltd. Mr. Beynon, whose new appointment became effective on December 1st, was formerly work study manager.

MR. A. S. D. BARRATT, technical director of Edwards High Vacuum, Ltd., since 1945, is resigning from the board of the company at the end of the year to become an independent consultant. His long association with the company will, however, be maintained by his appointment as technical consultant to the company, thus providing a liaison between the large scale developments of vacuum technology now taking place and the long-term developments of the Edwards international group of companies.

AT a meeting on November 18th, the board of directors of Northern Aluminium Co., Ltd., accepted the resignation of MR. C. P. PATON, who is taking up an important position with Aluminium, Ltd., in Montreal. At the same meeting, MR. TAYLOR CORNELIUS, who has taken over from Mr. Paton as Northern's general works manager, was elected a director of the company. Mr. Cornelius was until recently a vice-president of Aluminum Co. of Canada, Ltd., and manager of that company's fabricating division.

THE following changes in the responsibilities of senior executives of Union Carbide, Ltd., are announced: MR. L. E. WHITMORE becomes director and general manager, Polyethylene Division; MR. J. F. WIDMAN, previously director of marketing, chemicals, becomes director and general manager, Chemicals Division; and MR. R. WILSON has been appointed a director of Union Carbide, Ltd., and becomes director and general manager, Alloys Division.

MR. H. MORLEY has been appointed a director of Samuel Fox & Co., Ltd., a subsidiary of The United Steel Cos., Ltd. Mr. Morley, who is general works manager of Samuel Fox, joined the company in 1934. He became manager of the wire department in 1948 and chief work study officer of United Steel in 1954, returning to Samuel Fox in 1957 as works manager (services). He was appointed general works manager in 1958.

MR. F. A. ROSS (secretary), MR. W. G. A. JENKINS (personal assistant to the chairman) and MR. J. HIGGINBOTHAM (deputy general manager of the engineering department) have been appointed to the board of Edgar Allen & Co., Ltd.

MR. T. HAAS has been appointed head of the design section of the newly-formed members' service department of British Welding Research Association, Abington, Cambs. For the past six years, he has held the position of head of the fatigue department at the Bristol Aeroplane Co., Ltd.

AT a meeting of the board of The United Steel Cos., Ltd., last month, MR. H. P. FORDER and MR. A. R. HAY were appointed directors of the company. Mr. Forder is director and general manager of the Samuel Fox & Co., Ltd., subsidiary of the company, and Mr. Hay is a director of Naylor, Benzon & Co., Ltd., who represent United Steels in the Newcastle area.

SIR W. H. BAILEY & Co., LTD., (Albion Works, Patricroft, Manchester), announce that MR. A. KIRKHAM has resigned from the board of directors and joined the Foundries Division of David Brown (Industries), Ltd., Penistone.

IT is announced that ADMIRAL SIR MICHAEL M. DENNY, G.C.B., C.B.E., D.S.O., has been appointed a director of English Steel Corporation, Ltd.

THE Solartron Electronic Group, Ltd., have appointed MR. A. COCKLE as reliability engineer.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Low-Filtration X-ray Tube

In response to a number of requests for a low-filtration X-ray tube suitable for use with their MG 150 unit, C. H. F. Müller, Hamburg, have developed a completely new dual-focus tube and shield, rated at 150 kV., with a beryllium window. It is being marketed by Research and Control Instruments, Ltd., the sole distributors of Müller equipment in the United Kingdom. The tube is intended to meet a long-standing need in the plastics, rubber and light alloy industries, where a very low filtration tube is required if the best results are to be obtained. It is claimed that, at the present time, this is the only 150 kV. tube with a filtration of 3 mm. of beryllium available. The tube foci are 0·7 and 2·5 mm. and the maximum loadings on the MG 150 unit are 600 W. for the small focus and 1,800 W. for the large focus; the emergent beam angle is 40°. The distance between X-ray port and focal spot has been kept as short as possible, and a minimum distance of 70 mm. can be obtained between focal spot and the sample (lower edge of mechanical snap lock). By removing four screws and taking off the snap lock and baseplate, the focal spot can be brought to a minimum distance of 33 mm. from the sample (lower edge of the housing head).

With the 2·5 mm. tube focus and the maximum output of 1,800 W., the dose output ranges from 135,000 r./min. at 70 V. to 88,000 r./min. at 150 V. for a distance of 77 mm. focal spot; the corresponding outputs for a distance of 33 mm. focal spot are 640,000 and 420,000 r./min.

Research and Control Instruments, Ltd., Instrument House, 207 King's Cross Road, London, W.C.1.

Digital Voltmeter

In many fields of science and technology there is a need for fool-proof monitoring of physical quantities converted into voltages electronically to four figures. Such accuracy of indication is not usually possible by conventional methods of long-standing, and the Solartron Type LM 902 digital voltmeter has been designed to meet this demand. Like the three-digit voltmeter, Type LM 901, it is a transistorised instrument of lightweight, robust construction.

Dial readings over a range from zero to four figures are impossible to achieve, except by approximation by skilled operators on even the largest of dials or standard recording charts. Even then, when possible, mis-reading, parallax and statically or magnetically false readings are a frequent source of trouble. The LM 902 eliminates all the error factors of conventional meters, and presents the reading in inch-high, clear, unambiguous figures, which can be read from "across the room" in broad daylight by unskilled and semi-skilled personnel. The instrument is, therefore, of special value in such applications as production testing, the monitoring of dynamic process experiments, process control, and the permanent monitoring of system and instrumentation elements.

The figures are back-projected onto the front display as bright digits against a black (negative polarity) or red (positive polarity) background, or the measured voltage may be isolated or earth-referenced. There are five voltage ranges (0–0·1599 V. to 0–1599 V.) and the accuracy is better than 0·1%. There is also a variable dead-zone control for stabilising the reading on fluctuating inputs and up to 40 db. common mode rejection of 50 c.p.s., at the input. The range switch has a "Check Zero" position and two "Standardise" positions for the checking of the reference supplies for positive or negative signals and standardising against a built-in Weston standard cell.

The Solartron Electronic Group, Ltd., Thames Ditton, Surrey.

Level Controller

The Leveltron capacitance-operated level controller is designed to cover the majority of applications in industry for the control of free flowing solids and liquids, and for monitoring sight glass or manometer levels. A feature of the instrument is the continuously variable differential control, whereby site engineers can now choose from a wide differential range of 1–60 pf. by adjustment of two simple controls. The first sets the control point of the relay, and the second then varies the reset point without affecting the setting of the control point, and so fixes the differential as required. In this way it is possible to adjust the instrument to obtain the best conditions for each application, to make subsequent adjustments to meet changing plant requirements, and to use one such instrument where two of the fixed differential type would in many cases be necessary.

It is claimed that improved circuit design and careful layout are directly responsible for the rigid stability with which the high sensitivity is maintained, not only over time, but also with complete consistency from one instrument to another. Measurement is possible up to 160 yd. from the point of detection, and because the measuring unit is located in the main control box, and not in the electrode arrangement, there is no exposure

to vibration or to an injurious atmosphere, no loss of sensitivity with stability, and ease of access for maintenance. A reversible plug allows choice of direct or reverse acting mode of operation.

There are three basic instruments—general purpose, heavy industrial, and remote control models—each offering a choice of supply of 110, 240 or 440 V. A.C., and 40–60 c./s. A special-duty electrode is available for use with all types of powders and granular material—even those of low dielectric: it is also suitable for liquids of fairly high viscosity. Any deposit of material on one side of the electrode has no effect on its operation.

Thomas Industrial Automation, Ltd., Station Buildings, Altrincham, Cheshire.

Two New Magnesium Alloys

ZTY alloy

ZTY is a new magnesium-base wrought alloy developed by Magnesium Elektron, Ltd., for use at elevated temperature. It contains 0·75% thorium, 0·5% zinc and 0·6% zirconium and is available as sheet, plate, extrusions and forgings. The properties of ZTY alloy are developed without the need for heat treatment, and the low thorium content ensures that the alloy will be economically attractive compared with other high temperature wrought alloys of higher thorium content.

At room temperatures the 0·1% proof stress lies in the range 8·5–12·5 tons/sq. in.; ultimate tensile stress 15–18 tons/sq. in., and elongation 10–20%. Elevated temperature tensile tests show that the room temperature proof stress is retained up to 160–170° C. and the Young's modulus falls only slightly up to about 240° C. ZTY alloy possesses excellent short term creep characteristics up to about 350° C.

As with all magnesium-base alloys, forming should be carried out hot—in the temperature range 350°–400° C., depending on the severity of the deformation required. ZTY alloy is fully weldable by the argonarc process, with filler wire of the same composition, and the weld efficiency of at least 85% is retained at elevated temperatures. It may also be welded to the high temperature casting alloy ZT1 (D.T.D. 5005).

The corrosion resistance of ZTY alloy is similar to that of the other zirconium-containing magnesium-base alloys and it possesses the same ease of machinability.

MSR alloy

The latest addition to the M.E.L. range of casting alloys, MSR, is a fully heat treatable magnesium alloy based on the magnesium-rare earth metal-silver-zirconium system containing 2·5% silver, 2% fractionated rare earth metals, and 0·6% zirconium. This alloy was developed by J. Stone and Co., Ltd., and falls within the broad scope of British Patent No. 637,040 owned by Magnesium-Elektron, Ltd.; additional patent cover is being sought.

The alloy is at present available in two composition ranges, A and B, characterised by minimum elongation values of 4% and 2%, respectively, the main variation being in rare earth metal content. Test bars in MSR alloy show a 0·1% proof stress of 10–13·5 tons/sq. in.; an ultimate tensile stress of 15·5–18·5 tons/sq. in.; and an elongation of 2–10%.

In the temperature range 0–250° C., MSR alloy has the best 0·1% proof stress of all magnesium-base sand casting alloys, and shows better retention of Young's modulus with temperature than is usual with magnesium alloys. MSR shows markedly better short time creep strength than any other magnesium casting alloy at 200° C., and at 250° C., it remains the best alloy for times up to one hour and total strains of 0·5%. The new alloy has good long time creep strength (equivalent to that of ZRE 1 alloy) at temperatures of the order of 200° C. At 200° C. and 250° C. the fatigue endurance of MSR alloy for 50×10^8 cycles under rotating bending conditions, is higher than for any other magnesium casting alloy.

The alloy is stable at room temperature, but slowly over-ages on holding at 200° C. After 350 hours at this temperature the 0·1% proof stress falls by about 20%, but the tensile strength is only slightly affected. Prolonged testing has failed to reveal any tendency toward failure by stress corrosion.

Pressure-tight and complex castings are readily made in MRS alloy, which is fully weldable by the inert gas tungsten arc process. Anticipated uses include airframe castings requiring high strength at temperatures up to 200° C.; missile castings requiring high strength up to higher temperatures but for shorter times; and engine castings operating up to 250° C.: it may also prove useful for aircraft landing wheels.

Further details on these alloys may be obtained from two booklets recently issued by:

Magnesium Elektron, Ltd., Clifton Junction, Manchester.

Gas Cutting Developments

LOWER production costs and higher output are predicted as a result of two advances in the field of oxy-fuel gas cutting. The first of these developments is a system for profile machines using propane—Cutomation—which is claimed to be cheaper to run, faster, and more efficient than existing methods. The second is a hand-cutting torch operating on low-pressure propane, which requires no trimming of the fuel or oxygen control valves. The designers of the torch claim that propane starts and cuts as fast as any other fuel gas. Of American design, this precision equipment is to be manufactured in this country by British Industrial Gases, Ltd.

Cutomation increases cutting speeds by 30–50%, and reduces overall oxygen consumption by a fifth. A fast starting preheat flame reduces by 75% once the cut has

begun, with a consequent saving both in gas and time. No burning or rolling of the top edge of the cut occurs.

The faster speeds are due to the fact that the cutting oxygen stream expands within the nozzle, whereas such expansion normally takes place as the oxygen leaves the torch. In the new nozzle, the oxygen flows at supersonic speed and oxidises the metal in the form of a narrower kerf. This has the additional advantage that less metal is lost. Using propane as the fuel gas, a high quality cut is ensured, and less slag is formed at the bottom of the cut.

To convert standard machines to the Cutomation system, a pre-set regulator is required to ensure that the maximum amount of propane is drawn from the cylinder. A three-hose injector type torch is also necessary, the supplies being used for the centre cutting orifice, the fuel gas and the preheat oxygen. The torch is fitted with a high speed nozzle, and an additional regulator ensures that the fuel gas supply varies automatically according to the preheat oxygen pressure.

The hand torch is a standard torch for all fuel gases, different models in the range varying only in respect of the injector fitted. One such model (62-2) is designed for optimum results using low, medium or high pressure acetylene as fuel gas, while a second (62-2F) is for use with propane, butane and Pyrogas. Model 62-2N is for use with coal gas. Using propane, the 62-2F torch will draw up almost the whole of the cylinder contents. The nozzle employed makes possible faster starting and cutting, while up to 70% of fuel gas cost is saved.

An important advantage is that, when the correct propane setting on the regulator is used, there is no need to trim either the fuel or oxygen valves. This saves the operator time in adjusting valves, and eases ignition of the blowpipe when working outdoors. The torch also makes possible all forms of gouging with faster speeds than are obtainable with dissolved acetylene.

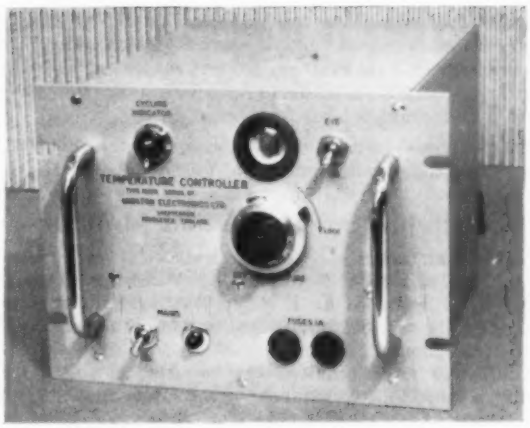
British Industrial Gases, Ltd., recently introduced to this country the gaugeless multi-stage regulator which, like Cutomation and the new hand torch, is manufactured by agreement with the Harris Calorific Company of Ohio. The equipment can be exported to all countries except Australia, South Africa and Canada.

British Industrial Gases, Ltd., 700 Great Cambridge Road, Enfield, Middlesex.

High Accuracy Temperature Controller

THE Winston-United Steel temperature controller Type M.226 is the outcome of co-operation between the laboratories of United Steel Cos., Ltd., and Winston Electronics, Ltd., in tackling the problem of the critical temperature control of furnaces used in creep testing alloy steels. It is claimed that this is the only commercially available instrument capable of controlling to such close limits as ±0·25° C. at 1,000° C., or above, for months on end. Fifty-six prototype instruments have been in continuous use for three years, with a total down-time for maintenance of three hours.

A platinum resistance thermometer is formed with an A.C. powered Wheatstone bridge, the preset arm of which consists of a 1,000 ohm. potentiometer graded in 4,000 steps, each being 0·25° C. The out-of-balance voltage from the bridge is amplified by a two-stage amplifier giving a gain of approximately 2,000. The degree of out-of-balance is displayed by a " magic eye "



indicator having two degrees of sensitivity. The amplified output is also fed to a phase sensitive bridge, which gives a positive or negative output according to whether the furnace temperature is lower or higher than the correct setting. This output is amplified 10 times by the D.C. amplifier and then applied to one section of the relay controlling the furnace relay. A time-interval generator provides switching cycles of normally 30 seconds to the other section of the relay control, which is so connected that the first section conducts, thus energising the relay only when the control signal from the thermometer is more positive than the signal from the time interval generator. When the furnace is at the correct temperature, the relay is energised for one half of the time cycle. The switching of the furnace relay is used to alter the furnace current between two values chosen to match the furnace characteristics. The fact that the furnace switching relay has no other function to perform avoids the critical adjustments of a normally troublesome component.

Winston Electronics, Ltd., Shepperton, Middlesex.

Duplex Plating Process

A NEW addition to the range of metal finishing interests held by Albright & Wilson (Mfg.), Ltd., is a duplex plating process that should be of considerable value to the automobile and allied industries. Last year the company introduced the Plusbrite process, which rapidly established itself as a bright nickel plating process that gave a fully bright deposit with better physical properties of plate than had hitherto been available. The new process is a logical development of the original Plusbrite process, and is designed to give maximum corrosion resistance together with a fully-bright plate, for such applications as motor car trim and similar automobile accessories.

A new additive is used, Plusbrite S.F., a sulphur-free addition agent which when added to the plating bath produces an electrolyte capable of giving a semi-bright deposit of high ductility and low internal tensile stress with excellent levelling properties: the deposit has a columnar structure. The standard Plusbrite solution is then used to produce a fully-bright finish. As the second deposit is lamellar in structure, a very high standard of corrosion resistance is attained by the duplex process. A

similar duplex process has been marketed in the U.S. since the early months of this year.

Plusbrite S.F. is very stable, and operating costs can be kept to a minimum during processing, as there is no loss by filtration through carbon.

Metal Finishing Department, Albright & Wilson (Mfg.), Ltd., 1, Knightsbridge Green, London, S.W.1.

Portable Contamination Monitor

E.M.I. ELECTRONICS, LTD., has produced a new compact all-transistor monitor for detecting radioactive contamination. Known as the E.M.I. Portable Contamination Monitor Type 1, the instrument employs the company's dual phosphor technique, and can be used for monitoring radioactive contamination on benches, clothing, and similar objects. All-transistor circuitry enables the instrument to be operated for long periods from a battery supply.

The dual phosphor technique enables both *alpha* and *beta* contamination to be monitored simultaneously. Separate distinctive tones are provided for *alpha* and *beta*, either on headphones worn by the operator, or on a built-in loudspeaker. The new monitor provides high accuracy over a wide temperature range (0–45° C.), and by using a single switch it is possible to monitor the sum of *alpha* and *beta* contamination, *beta* contamination alone, or just *alpha* contamination. This information, in the form of counts per second, is displayed on a meter covering the range 0–5,000. A high-pitched tone indicates *alpha* contamination and a low tone *beta*. Both notes are heard when *alpha* and *beta* are monitored. When the type of contamination has been identified, an accurate check can be made on either the *alpha* or *beta* positions. For monitoring very low *alpha* activities an electro-mechanical register is included to give a more accurate display at rates below about 6 per second.

E.M.I. Electronics, Ltd., Hayes, Middlesex.

Polarographic Instruments

THREE polarographic instruments designed at Professor Heyrovsky's Polarographic Research Institute in Prague, which are exported by KOVO, the Czechoslovakian state import/export organisation for the Scientific Instrument Industry and are already widely used in Eastern Europe, are now sold and serviced in the U.K. by the sole agents, Nash & Thompson, Ltd.

The KOVO P.576 polaroscope is an oscilloscope specially adapted to polarographic analysis. The method is based on the principle of an electrode system through which is allowed to pass an A.C. of 50 c/s. from a 300 V. power supply of high internal resistance; the polarisable electrode is charged during one cycle of the A.C. within a potential range from 0 to −2 V., and then back again from −2 V. to 0 (depending on solution composition). The electrode process is shown on the C.R. tube, where the course of the potential derivation with regard to time (vertical direction) is shown depending on the electrode potential (horizontal direction).

The results can be observed immediately after adjusting a suitable intensity of the A.C. current, the D.C. component and setting the required size, brightness and sharpness of the picture. The oscillogram shown on the C.R. tube is oval in shape for a pure basic electrolyte, the upper half representing the cathodic, and the lower half the anodic process. Following the introduction of a

depolariser into the electrolyte, the curve shows incisions in both anodic and cathodic parts. The depth of the incision is proportional to concentration, and its position is characteristic of the depolariser. The mutual position of the cathodic and anodic incisions is a measure of the reversibility of the electrode process. If they are situated at the same potential, the process is reversible; if displaced horizontally, or if one of them is absent, the process is irreversible.

The instrument is suitable for analysis in the range 10^{-3} M. to 5×10^{-5} M. Concentrations down to 10^{-9} M. can be reached by electrolytic concentration (turning the "Micro" switch on the instrument) or by a hanging drop method. Comparative titrations can be performed by using twin electrode systems and observing two oscillograms displaced horizontally. Titrant is added until the depth of incisions coincide. Isomers and compounds of similar structure can be very easily examined and identified.

The KOVO LP.54 manual polarograph is an elegantly styled conventional D.C. polarograph with one unusual feature which makes it semi-automatic. A recorder is built into the instrument, and light reflected from the galvanometer shines on the nearly transparent paper from behind. The knob which increases the voltage across the electrolyte cell also advances the recorder paper; one can thus plot the polarogram directly on to the paper.

The KOVO LP.55A polarograph is a photographic recording instrument of a type not manufactured in the U.K. A sensitivity of 0·1 μA. full scale deflection is obtained by suspending its galvanometer on a cantilever 3 ft. above the instrument. Operation is motorised and fully automatic, once the voltage range that it is required to scan has been set on the instrument. Automatic recording of abscissae after every 100 or 200 mV. is performed, so that the photographic record can be easily evaluated.

Nash & Thompson, Ltd., Hook Rise, Tolworth, Surbiton, Surrey.

CURRENT LITERATURE

Book Notices

NON-FERROUS METALS

STATISTICS 1958—TREND 1959

125 pp., numerous illustrations and tables. Published by the Organisation for European Economic Co-operation and available from H.M. Stationery Office. 10s.

A SPECIAL supplement prepared by the O.E.E.C. Non-Ferrous Metals Committee at the end of October, and dealing with market developments during the first nine months of 1959 and the prospects for the non-ferrous metals industries in the near future, brings up to date the committee's annual statistical report, which covers, in general, statistics for 1958 and trends for 1959.

As in previous years, the main body of the report consists of detailed statistical information for the O.E.E.C. countries as a whole, and for each individual member country; it covers production, consumption, trade and uses at the first processing stage for aluminium, copper, lead, zinc, tin and nickel. Information on uses at the first processing stage is provided over a period of five years, and is of special interest to producers of semi-finished products and castings. The statistical tables are prefaced by observations on the trend of the market for each metal during the period covered. Graphs of the trade pattern of aluminium, zinc and tin show the development of imports from Eastern countries.

A new series of statistics gives figures for aluminium end-uses in Europe during 1958. Thus, for the first time, the report provides a break-down, on a comparable basis, of the flow of processed aluminium to consumer industries such as vehicle production, mechanical and electrical engineering and packaging.

GLOSSARY OF FOUNDRY AND METALLURGICAL TERMS

ENGLISH-FRENCH, FRENCH-ENGLISH

28 pp., 4½ × 5½ in., paper covers. Published by The British Cast Iron Research Association, Alvechurch, Birmingham. 7s. 6d. or $1·10.

THIS little glossary, based on the experience of the B.C.I.R.A. Information Department for a number of years, aims to supply foundrymen and metallurgists with English equivalents of French terms commonly used in the literature of ironfounding and ferrous metallurgy. It is, in fact, a working vocabulary and the terms included have been carefully examined in relation to their original context.

Trade Publications

THE efficient treatment of quenching oil has for a long time been a problem in the heat treatment of metals. Circulation of the oil through a suitable cooler keeps the temperature down, thus reducing the tendency to form vapour bubbles, with consequent loss of quenching efficiency, and reduces the liability to excessive oxidation. The problem of cooling quenching oil is discussed in a new brochure, No. 591, issued recently by The Visco Engineering Co., Ltd., which features the Visco Spray-blast cooler, the first oil cooler to use a mixture of air and water as the cooling medium, instead of air only or water only, as had been the previous practice. The system incorporates the advantages of both, without their disadvantages. These coolers can, of course, also be used for cooling transformer oil, etc.

WE have received from Crofts (Engineers), Ltd., Bradford, a copy of Publication No. 5938, entitled "Steel Castings." The available facilities enable the production of castings varying from a few ounces to 7 tons, and repetition work is a speciality. Large up-to-date machine shops are capable of machining the most complex casting to precision limits. The brochure features illustrations of some of the large castings in the range made by the company.

SINCE the announcement of the cessation of publication of *The Craven Machine Tool Gazette* at the end of last year, and its replacement by another house journal, evidence of its popular appeal to machine tool users all over the world has led to a reconsideration of the decision. The new form of the journal will be less voluminous than formerly, but it is hoped to issue a new number about every two months, instead of three or four times a year. The first issue—September-October 1959—features copying equipment, in one case as applied to a 36 in. centres sliding, surfacing and screwcutting lathe, and in the other to a 16 ft. diameter vertical boring and turning mill.

FLEXIBOX LTD., have produced a new comprehensive mechanical seal catalogue in which is described the full range of Flexibox balanced and unbalanced mechanical gland seals for all types of rotary shaft equipment—pumps, mixers, agitators, compressors, etc. This catalogue has been primarily designed to provide mechanical seal users with a rapid means of selecting the correct type and size of seal for any given application. Copies of the new catalogue are obtainable from Public Relations Department, Flexibox, Ltd., Nash Road, Trafford Park, Manchester, 17.

THE September October issue of *The Nickel Bulletin* contains more than a hundred abstracts of technical literature pertaining to nickel and nickel-containing materials. The diversity of the subjects covered provides an apt illustration of the wide range of industries and applications in which nickel plays a major rôle. In the section concerned with heat- and corrosion-resisting materials, particular attention may be called to a group of abstracts covering literature on the joining of austenitic to ferritic steels. The range of the other items may be indicated by mention of some of the fields concerned: methods of hardening nickel-base alloys; the relationship between stabilising additions and creep-resistance; factors influencing, and evaluation of high-temperature properties; use of austenitic steels in power plants; properties and applications of corrosion-resisting steels and alloys. Abstracts of papers reporting investigation of the properties and fabrication of ultra-high-strength constructional steels exemplify the growing interest which these materials are arousing in connection with aeronautical applications, while an item of considerable

interest in the section on cast irons refers to a comprehensive evaluation of the suitability of alloy irons for use in crushing and grinding plant. Nickel and its compounds are represented in references to papers on extraction, properties and analysis. Literature abstracted in the other sections of the issue ranges in subject matter from the properties of non-ferrous alloys and permanent-magnet materials to the operation of nickel-plating solutions and the influence of plating variables on the coatings obtained. The issue also contains the usual quarterly review of recent patent literature.

A 25-PAGE illustrated book on marine paints and finishes has been published by Jenson and Nicholson, Ltd., paint and varnish manufacturers, of Stratford, London. It is intended as a quick reference to all types of marine finishes and their application. It is the purpose of this book to help the ship-owner by presenting a brief summary of the problems that affect him and of the best answers to them. Subject matter includes a systematic review of the paint needs of the different parts of a ship; corrosion, fouling and weathering; liquid envelope—a new protective covering; and types of marine paint.

SOME useful notes on "Suggested Applications of Rocol Molybdenised Lubricants for Machine Tools" have been produced by Rocol, Ltd., Rocol House, Swillington, Leeds, and General Buildings, Aldwych, London, W.C.2. The general applications of Rocol molybdenised lubricants to beds and slides, lead screws, control valves, charge wheels and open gears and thrust washers are indicated. More specific recommendations are made for a number of machines—grinding, boring (including jig borers), planing, radial drilling, shaping, milling, lathes and gear cutting, involving the wide range of Rocol molybdenised lubricants, which provides the means by which the useful life of machine tools and components can be increased and precision maintained. Recommendations by some machine tool makers for the use of Rocol products in their designs are indicated.

THE latest edition of *Wiggin Nickel Alloys* features uses of Wiggin products ranging from an advanced electrical furnace to sea water evaporators. The aircraft industry is featured in articles on the Conway-powered Boeing 707 and the power behind the Thor, Jupiter and Atlas rockets. A further notable contribution is an article on the use of the Brightray series of nickel-chromium electrical resistance materials in pre-heating for welding and stress-relieving of large vessels. *Wiggin Nickel Alloys* is obtainable free on request from Henry Wiggin and Co., Ltd.

ALTHOUGH magnesium is being used today in a very wide range of industries, its full potential is unexploited, and one of the principal aims of the Magnesium Industry Council is to help people to get to know more about the metal; to tell them where it can be used to advantage; and to show them how to use it in the most economical way. The reception accorded a 28-page publication *This is Magnesium* last year has encouraged the Council to decide to publish further information from time to time under the same title. The first issue in this new form gives details in support of the claim that magnesium is the world's fastest machining metal, and show, with illustrations, a number of typical applications. Copies may be obtained from the Council at Dickens House, 15 Took's Court, London, E.C.4.

THE third issue of *Die Casting News*, published by the Zinc Alloy Die Casters Association, is an eight-page bulletin containing information, with photographs, on contemporary applications of zinc alloy die castings. The parts have been chosen to illustrate particular advantages of die casting in zinc alloy, such as the ability to produce complex shapes in one fast operation, the elimination of laborious time-consuming machining, and the ease with which fastening devices, bearings, and so on, can be cast in place. In addition to these functional assets, the fine smooth surface of zinc die castings enables highly decorative and durable finishes—enamel or chromium—to be easily applied, and examples in this issue include a shaver head, medical lamps and a T.V. set bezel. Copies are available from the Zinc Development Association, 34 Berkeley Square, London, W.1.

THE advantages of cold rolled sections for metal fabrication work in a wide variety of industries and the production facilities available at the company's modern plant are described in a new illustrated brochure issued by Warwick Rim & Sectioning Co., Ltd., Gold Green, West Bromwich, Staffs. The brochure shows how complex shapes can be produced to very close limits, enabling the designer to avoid using separate components by incorporating them in one section. It also shows how composite sections, using dissimilar materials, can be produced. Various applications of the process are illustrated and details are given of ancillary services which are available.

Books Received

"Handbook of Practical Electroplating." By T. M. Rodgers. 333 pp. New York and London, 1959. The Macmillan Company. 59s. 6d.

"High Temperature Materials." Edited by R. F. Hehemann and G. Mervin Ault. Based on a Conference held in Cleveland, Ohio in April 1957, and sponsored by the High Temperature Alloys Committee, Institute of Metals Division, The Metallurgical Society, American Institute of Mining, Metallurgical and Petroleum Engineers. 544 pp. inc. index. New York and London, 1959. John Wiley & Sons, Inc., and Chapman & Hall, Ltd. 140s. net.

"The Physical Metallurgy of Rolling," by Dr. F. H. Scott. 94 pp. inc. index. Devon, 1959. Arthur H. Stockwell, Ltd. 10s. 6d. net.

"Corrosion of Chemical Apparatus—Corrosion Cracking and Methods of Protection Against It." By G. L. Shvartz and M. M. Kristal. Translated from Russian. 250 pp. New York and London, 1959. Consultants Bureau, Inc., and Chapman & Hall, Ltd. 60s. net.

"Crystal Growth." Discussions of the Faraday Society, No. 5, 1949. 366 pp. inc. author index. London, 1959. Reprinted by Butterworth's Scientific Publications. 60s. By post 2s. 0d. extra.

"Physico-Chemical Measurements at High Temperatures." Edited by J. O'M. Bockris, J. L. White and J. D. Mackenzie. 394 pp. inc. author and subject indexes. London, 1959. Butterworth's Scientific Publications. 75s. By post 2s. 3d. extra. The American edition of this book is published by Academic Press, Inc., New York, at $13.50.

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NOVEMBER, 1959. Vol. LX, No. 361

The Determination of Sulphur in Cast Iron

A Study of the Combustion Method with an Assessment of the Value of Vanadium Pentoxide as Flux

By H. Green

In the determination of sulphur in cast iron by the combustion method, recovery of this element is not stoichiometric. In this paper the author discusses the results of an investigation into the possible sources of loss and the effect of using fluxes, in particular vanadium pentoxide. He concludes that to obtain anything approaching stoichiometric recovery it is necessary to find some means of retaining the sulphur trioxide formed and to complete the determination by an alkali titration.

IN determining the sulphur content of cast iron samples by combustion, a number of variations in the conditions used are made by individual analysts, but the basic procedure remains the same. A suitable sample weight is ignited in a controlled stream of oxygen and at a controlled temperature. Oxides of iron, etc., are filtered out of the gas stream by means of refractory, asbestos or cotton wool plugs. The gas issuing from the end of the combustion tube then consists of a mixture of the excess oxygen, carbon dioxide, sulphur dioxide and sulphur trioxide. This mixture of gases is passed through a suitable absorbent and the solution titrated with a standard solution such as iodine, borate or hydroxide. By ignition of standard cast irons of known sulphur content a "recovery factor" is obtained; this is then used to calculate the percentage of sulphur in the samples. The determination and use of a recovery factor is necessary because the recovery of sulphur is not stoichiometric; it is usually in the region of 85–90%.

Possible Sources of Error

An assessment of the individual sources of error in the combustion method for the determination of sulphur in steel was published by Smith, Backhouse and Woodward.[1] Using a furnace temperature of 1,300° C. and an oxygen flow of 3 litres/min., they gave the following figures:—

Sulphur evolved as SO_2	86%
Sulphur as SO_3 absorbed with SO_2	3%
Sulphur as SO_3 escaping absorption	1%
Sulphur as SO_3 condensed in apparatus	4%
Sulphur retained in residue	6%
	100%

In the case of cast iron, in general 85–90% of the sulphur appears to be evolved as the dioxide when the determination is finished by titration with iodine and thiosulphate. If the finish is by titration with alkali, the recovery of sulphur appears to be just a little better.

Condensation in the Apparatus

To reduce condensation of sulphur trioxide to a minimum, it would seem advisable to reduce the distance between the end of the combustion tube and the absorbent as far as possible. This, in practice, is what is normally done. So to test this it was decided to increase the distance from combustion tube to absorbent by inserting 10 ft. of tubing. It was found that the recovery of sulphur as the dioxide remained constant. The only noticeable point was the complete absence of the usual white fumes when using the longer train. It appeared, therefore, that any condensation taking place did so very soon after the gases left the hot zone of the furnace, and no further conversion of sulphur dioxide to sulphur trioxide would appear to take place when the gases pass out of the furnace. It was felt that, since the gases are normally "filtered" by some type of porous plug, on which oxides of iron generally collect in greater or lesser quantities, absorption or adsorption might be taking place there.

Experiments were carried out where the sample was given a pre-heat period in the hot zone of the furnace before passing the oxygen. In this way the incidence of iron oxide was reduced to a minimum and the filter could be dispensed with under these circumstances. The yield of sulphur was not improved to any noticeable extent.

Sulphur Retained in the Residue

There is claimed to be evidence that some sulphur is retained in the residue after the combustion has been carried out. This seems to be based mainly on the fact that higher yields of sulphur are obtained by the use of higher combustion temperatures. It does not follow, however, that the better yield of sulphur is due to recovery from the residue. The reaction for the oxidation of sulphur dioxide to sulphur trioxide is exothermic, approximately 32,000 calories being liberated for each gram molecule of the sulphur oxidised from the dioxide to the trioxide. It follows from this that, if the temperature were raised sufficiently, the concentration of sulphur trioxide might be reduced to a level such that it could be ignored. The major difficulty associated with using such a procedure lies in the fact that the gases would need to be cooled again before being absorbed, and this would make

TABLE I—EXPERIMENTAL RESULTS

Sulphur Content of Sample (%)	Oxygen Flow (litres/min.)	Temperature (° C.)	Titration (ml.)	S Recovered as SO_3 (%)
0·259	1	1,350	23·90	72·1
0·259	1	1,350	23·90	72·1
0·259	2	1,350	26·40	79·8
0·259	2	1,350	23·30	70·3
0·259	2	1,150	26·65	80·6
0·259	2	1,150	25·20	76·2
0·259	1	1,150	27·20	82·3
0·259	1	1,150	27·40	82·9
0·113	1	1,150	11·35	76·5
0·113	1	1,150	11·35	79·5
0·113	2	1,350	9·70	64·8
0·113	2	1,350	8·45	56·0
0·113	2	1,350	9·60	64·0
0·113	2	1,350	9·60	64·0
0·113	1	1,150	11·00	74·0
0·113	1	1,150	10·40	69·8
Blank	1	1,350	0·55	
Blank	2	1,350	0·55	
Blank	2	1,150	0·55	
Blank	1	1,150	0·55	

1 ml. of titrant = 0·008% S on a 1 g. sample

possible the reverse reaction. To minimise the extent to which this occurred, very rapid cooling, combined with rapid transfer of the gases from combustion tube to absorbent, would be necessary.

This, in turn, would introduce another possible error. Rapid gas flow might carry some of the sulphur dioxide through the absorbent and permit its escape into the atmosphere—even when a sintered bubbler was used to break the gas stream. It is also of importance to bear in mind that ferric oxide, at temperatures exceeding 600° C., catalyses the reaction of sulphur dioxide and oxygen.[2] If such iron oxides were swept along the tube and deposited on the walls, the surface area exposed could have a not inconsiderable catalytic effect. The other point to note is that the greater oxygen flow would have a cooling effect which would in turn call for higher furnace temperatures.

Sulphur Trioxide Escaping Absorption

It is frequently stated that the reason for low sulphur yields, even using an alkali titration, is the fact that a part of the sulphur trioxide, which it would appear is formed, is in a condition where it passes through the absorbent and escapes into the atmosphere. The white fumes seen with most samples are reputed to consist of a mixture of sulphur trioxide and water vapour. It is interesting to note that two samples of the same iron, ignited consecutively without variation in any of the experimental conditions, not infrequently give vastly different quantities of white fumes; while the recovered amount of sulphur is the same. Further than this, white fumes are obtained with samples containing very low percentages of sulphur, and on occasion malleable irons with sulphur contents above 0·2% give practically none, all other conditions being constant. It would seem from this that, despite rigidly controlled conditions, the physical condition of any sulphur trioxide which is formed is not always the same.

A consideration of the foregoing can now be made.

(1) No method has yet been reported which claims to obtain all the sulphur from a sample of cast iron as sulphur dioxide when the sample is burnt in a stream of oxygen. This is due to the fact that optimum conditions for the variables of the method react against one another. It is considered that, of the methods in general use, only an alkali titration could hope to show anything approaching 100% recovery of sulphur.

(2) Because of the risk of absorption, adsorption or catalysis by ferric oxide, it is advisable to eliminate this as far as possible (see "Use of Fluxes"). When this has been achieved it is then possible to dispense with the filter plug, which will eliminate the build-up of pressure in the combustion tube and allow conditions to be standardised.

(3) The evidence is contradictory as to whether any of the sulphur present in the sample could remain behind in the residue at the temperature attained during combustion. There is reason, therefore, to take such an obvious precaution as raising the temperature of the furnace to, say, 1,500° C., particularly as this may help to keep the concentration of sulphur trioxide to a minimum.

(4) The rate of oxygen flow has never appeared to be a very critical factor in the influence it exerts on the sulphur recovery. Rates of flow between 250 and 3,000 ml./min. have been recommended by various workers on various grounds. Experience has shown that the rate of flow, providing it falls between these limits, is not so important as that it shall remain as constant as possible. An oxygen flow of 750 ml./min. is at present in use in the laboratories of the B.C.I.R.A., but flow rates from 750 ml./min. to 1,500 ml./min. are reasonable to keep the cooling effect as low as possible.

(5) All operators of combustion sulphur methods agree that the distance between the end of the combustion tube and the absorbent should be kept to a minimum. It is advisable to adhere to this practice, although no loss of sulphur dioxide appears to result from the use of a longer train between combustion tube and absorbent. The use of a sintered bubbler to give maximum surface area of contact between the issuing gases and the absorbent is obviously advantageous.

(6) It has always been a question as to whether samples should be fluxed or not. This problem is so closely related to the question of preheat period and reduction of the formation of iron oxides that it was considered necessary to carry out experiments to determine the place that fluxing takes in the problem as a whole.

Use of Fluxes

Reduction of the amount of iron oxide carried along in the gas stream was felt to be the first step to improving the combustion sulphur method. This would mean that it would be possible to dispense with the filter plug. Despite the fact that some operators claim that a two or three minute preheat period makes it possible to ignite samples of cast iron without iron oxide carry-over, complete success was not achieved. A considerable improvement was noticed, but small quantities of oxide began to appear at the sides of the tube and in the absorbent after only a small number of tests. Prolonging the preheat time did not have any effect. It was decided, therefore, to try a number of fluxes and compare their action. At the same time it was thought that it would be interesting to compare the action of (*a*) fluxes which contain no available oxygen; (*b*) reagents containing oxygen but which do not melt at the temperature used; and (*c*) fluxes which contain available oxygen.

In the first group—fluxes containing no available oxygen—are the metals with low melting points, such as tin and lead. Experiments soon showed that these were

not going to prove helpful. The yield of sulphur was in general lower than before, iron oxide was still not completely eliminated, even after three minutes preheat, and the tube was rapidly covered with slag at the point directly above the combustion boat. Further than this, it was necessary to have a by-pass for the oxygen, or to isolate the combustion tube and absorbing vessels from one another, otherwise the partial vacuum created in the tube during the preheat caused the absorbent to suck back violently.

In the case of the reagents which do not flux at the temperature of combustion, such as manganese dioxide or calcium oxide, the result was that iron oxide mixed with some of the reagent was carried along the gas stream. The use of such chemicals had to be abandoned.

Considerably more success was achieved with reagents which contain oxygen and also flux. A number of such chemicals were tested, including lead dioxide, lead monoxide and vanadium pentoxide. The best results were obtained with the vanadium pentoxide and are quoted here for the useful information which they give. In a test series, using a temperature of 1,300° C. and a 1 g. sample of cast iron, amounts of vanadium pentoxide in 0·5 g. increments from 0·5–3·5 g. were used with preheat periods from 0·5–10·0 minutes in the hot zone before passing the oxygen at 750 ml. min. It soon became apparent that 3 minutes preheat was the minimum period necessary to avoid carry-over of iron oxide or powdered vanadium pentoxide. Also that one could preheat indefinitely and still get iron oxide unless the amount of vanadium pentoxide exceeded 2 g. From 2·5 g. upwards no trouble was experienced with three or more minutes preheat. It appears that the sample must be partially pre-oxidised in the furnace by the vanadium pentoxide. Assuming the sample of cast iron contained 4% carbon, 2% silicon and 94% iron, it was calculated that 0·54 g. of oxygen was required for complete oxidation to CO_2, SiO_2 and Fe_2O_3.

In the decomposition of vanadium pentoxide, 182 g. provide 32 g. of oxygen. Thus 0·54 g. of oxygen is obtained from just over 3 g. of pentoxide. Allowing for the oxygen already in the tube, it would appear that about the theoretically required amount for complete oxidation of 1 g. of cast iron would be present. It was hoped that this would mean that the sulphur would be converted mainly to the dioxide, and that this could be swept into the absorbent in the usual way.

It was immediately apparent that this was not the case, when a few samples were ignited and titrated. It has been stated that vanadium pentoxide can be used as a catalyst in the conversion of sulphur dioxide to sulphur trioxide, and this may have been causing the trouble. Certainly when a series of standard samples, fluxed with 3 g. of vanadium pentoxide, was given pre-

TABLE III.—SUMMARY OF THE ANALYSIS OF VARIANCE TABLE

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square
Between S levels	288·15	1	288·15
Between T levels	385·14	1	385·14
Between F levels	29·45	1	29·45
$S \times T$ interaction	33·35	1	33·35
$S \times F$ interaction	17·43	1	17·43
$F \times T$ interaction	22·80	1	22·80
$S \times F \times T$ interaction	5·64	1	5·64
Total between groups	781·94	7	
Experimental error	106·45	8	
Total variation	888·39	15	

TABLE II.—ANALYSIS OF VARIANCE TABLE

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square
Between sulphur levels (S)	288·15	1	288·15
Between temperature levels (T)	385·14	1	385·15
$S \times T$ interaction	33·35	1	33·35
Between groups	706·64	3	
Between flow rates (F)	29·43	1	29·43
Between sulphur levels (S)	288·15	1	
$F \times S$ interaction	17·43	1	17·43
Between groups	335·01	3	
Between T levels	385·14	1	
Between flow rates	29·43	1	
$F \times T$ interaction	22·80	1	22·80
Between groups	437·3		

heat periods of three to fifteen minutes, a progressively lower yield of sulphur as the dioxide was obtained.

Another series of samples treated as above, but with 0·25 g. of calcium hydroxide added to the 3 g. of pentoxide, only served to show that the presence of moisture (derived from the calcium hydroxide) gave very low yields indeed of sulphur dioxide. This was to be expected since it is well known that the presence of water vapour will catalyse the oxidation of sulphur dioxide to sulphur trioxide. It emphasises the need for using dry oxygen and dry vanadium pentoxide in all combustion sulphur determinations. The vanadium pentoxide should be dried for two or three hours at 110° C., and the oxygen dried by passing it through a prolong tube containing anhydrous magnesium perchlorate.

At this stage it was decided that, using 3 g. of vanadium pentoxide as flux, dried as above, a series of tests should be carried out and a statistical analysis made of the results obtained. In this series two furnace temperatures were used, 1,150° C. and 1,350° C.; two oxygen flow rates of 1,000 ml./min. and 2,000 ml./min., and two British Chemical Standards containing 0·113% and 0·259% sulphur. Blank determinations were carried out on the pentoxide at both temperatures and oxygen flow rates. In each case the absorbent contained excess iodine: the amount of iodine used was determined by back titration with thiosulphate using starch as indicator.

The results obtained (Table I) were studied by the technique of analysis of variance (Tables II and III).

From these results, applying a statistical test, it can be found that only the sulphur content variations and temperature variations caused a significant change in efficiency. This does not mean that the other factors have no effect, only that they are too small to be detected by these experiments.

One or two other points arise from these experiments. It becomes obvious that at higher temperatures more sulphur trioxide is formed; this may be caused by the catalytic action of the vanadium pentoxide. The lower the percentage of sulphur present, the more this becomes obvious, as the proportion of trioxide is greater. The overall yield of sulphur as the dioxide is quite considerably lower using vanadium pentoxide as flux. The proportional yield of sulphur as dioxide is roughly constant for the two rates of flow of oxygen: this is to be expected, since the samples are preheated for three minutes with a fairly constant volume of available oxygen in a confined space.

Conclusions on Use of Vanadium Pentoxide as Flux

Despite variation of temperature and oxygen flow rate over wide ranges, it is not possible to hope to recover

all the sulphur from a cast iron by any combustion method which does not employ some means of recovering both sulphur dioxide and sulphur trioxide. Condensation loss of sulphur, as trioxide, in the absorption train is likely to be at a minimum providing that the distance between the combustion tube and the absorbent is kept to a minimum. It does not seem probable that any appreciable quantities of sulphur remains behind in the residue, after three minutes preheat and three minutes oxygen sweep, at the high temperatures normally used.

The use of vanadium pentoxide as a flux eliminates both the carry-over of iron oxides and the creation of a partial vacuum in the combustion tube, providing sufficient flux is used.

A statistical study has been made of the combustion sulphur method using a 1 g. sample fluxed with 3 g. of vanadium pentoxide and a preheat period of three minutes. The determination was completed by iodine titration to obtain the percentage yield of sulphur as the dioxide. The rate of oxygen flow and the temperature of combustion were varied for two ranges of sulphur contents. It was found that the yield of sulphur as sulphur dioxide using these conditions was lower than by the more usual combustion methods, possibly due to the catalytic action of the vanadium pentoxide. The ratio of sulphur dioxide and sulphur trioxide was independent of the rate of oxygen flow in the range 1–2 litres/min., but dependent upon both the percentage of sulphur present and on the temperature of combustion.

To obtain anything approaching stoichiometric recovery of sulphur from cast iron using a combustion method it is necessary to find some means of retaining the sulphur trioxide, which it appears is inevitably formed, and to complete the determination by alkali titration. In such circumstances vanadium pentoxide would be very valuable as a suitable flux to eliminate the incidence of iron oxides in the gas stream.

Acknowledgments

The author wishes to thank the Council and Director of the British Cast Iron Research Association for permission to publish this paper. Also Mr. W. Oldfield, who carried out the mathematical examination of the results obtained.

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2 Lowry, T. M., "Inorganic Chemistry", 2nd edition 1946, Macmillan & Co. Ltd., London, pp. 381-382.

New Furnace for N.P.L.

THE General Electric Co., Ltd., in association with Vacuum Industrial Applications, Ltd., have received an order from the National Physical Laboratory for a 2-litre vacuum arc-melting furnace. The furnace will be used for melting and casting refractory metals such as titanium, niobium, molybdenum, etc. The furnace may be used for melting by either the consumable or non-consumable electrode process. In the former case the ingot is formed from an electrode of the parent metal which is fed progressively into a molten pool in the mould; non-consumable electrode melting is effected by using a tungsten electrode to strike an arc into which the charge is fed as small particles.

The power is being supplied by a 2,000 A., 40 V. germanium rectifier unit, whose output is controlled by means of a saturable reactor connected in the primary side of the rectifier transformer, enabling the output current to be varied from 500 A. up to 2,000 A. The power unit has a short time overload capacity of 2,500 A. A two-stage pumping system is provided comprising an oil booster-diffusion pump backed by a rotary pump. The working chamber pressure is 0·1 microns Hg. and the pumping speed is sufficient to maintain this pressure against gas evolution from the electrode and molten pool.

Radiation Monitor Order

THE United Kingdom Atomic Energy Authority has recently placed a contract with E.M.I. Electronics, Ltd., for 43 hand and clothing monitors, which brings the total number sold to nearly 100 since it was first announced less than a year ago. Orders have already been received from Germany, Belgium, Israel and Australia. The E.M.I. monitor, part of the Emirad range of nuclear health instruments, has the unique feature of being able to monitor both hands simultaneously for *alpha* and *beta* contamination, thus reducing the hand count to five seconds, and at the same time checking two other person's clothing and footwear. Automatic compensation for background radiation means that monitors can be sited on the most convenient positions, regardless of nearby radioactive materials.

Steelworks Instrumentation in Spain

MR. W. SWINN, steel industry sales manager of Honeywell Controls, Ltd., recently made a tour of Spanish steel centres, where a paper by him on instrumentation of furnaces in the steel industry was read by Señor Manuel Doblas, representing the local Honeywell distributor. It is believed that this is the only paper on furnace instrumentation printed in Spanish. Apart from the lectures, Mr. Swinn supervised demonstrations of liquid steel temperature measurement in the plants of individual firms.

New Detergent Company

THE formation of Reddish Detergents, Ltd., Stanley Road, Cheadle Hulme, Cheshire, to take over certain commitments previously handled by the Reddish Chemical Co., Ltd., is announced. Reddish Detergents, Ltd., will have a range of detergents for the metal industries. In addition, it will specialise in the manufacture of products for firms who wish to market their own formulations.

Griffin & George Branch

THE first step in the policy of greatly improving their national coverage was made last month by Griffin and George, Ltd., when their new branch was opened at 626 Welbeck Road, Walker, Newcastle upon Tyne. To mark the occasion two exhibitions were held—one in Newcastle upon Tyne and the other in Middlesbrough—showing how the resources of the group are maintaining and improving the service to laboratories of all kinds.

The Preparation of Lead Cable Sheath Materials for Microscopical Metallography*

By P. Gregory, A. J. Bangay and R. Paris

The preparation for microscopical examination of lead cable sheath alloys presents certain difficulties. Details are given of techniques developed in the authors' laboratory for the mounting, grinding, polishing and etching of such materials, and the quality of the results achieved is indicated in a series of photomicrographs.

THE microscopical examination of metals and alloys depends, to a very large extent, on the ability to prepare a suitable surface which will reflect an image of the structure. Various methods are available which achieve this object, and many improvements have been developed in recent years, the most notable being electrolytic dissolution.[1] Methods for lead materials, however, appear to have received little attention beyond brief mention in standard textbooks on the subject.[2, 3] The present paper gives an account of the techniques which have been developed in this laboratory in the course of extensive experience with lead cable sheathing alloys.

The standard method utilises abrasive and chemical polishes and, except for some minor refinements, follows closely the procedure evolved by Worner and Worner.[4] It should be mentioned that its success requires a strong element of personal skill, particularly in the finishing stages. An attempt to overcome the human element by the development of an electrolytic dissolution technique has yielded promising results,[5] but the method appears to have certain limitations restricting general applicability.

Mounting

When examination at magnifications higher than × 50 is required, it is usually desirable to mount the specimen in a cold setting resin to facilitate the high standard of preparation necessary. N.H.P. mounting medium†[6] and araldite D‡, acrylic and epoxy resins, respectively, have been found to be suitable for this purpose. The first material has the advantage of giving a mount within 20 minutes; the second requires about 24 hours to set at room temperature but gives more satisfactory results. In both cases, the liquid resin is poured around the specimen resting on a glass plate and contained in a brass ring section, the surfaces being previously greased to prevent adhesion of the resin.

Hot pressure moulding, in substances like Bakelite, should never be employed for lead materials, as the temperatures involved are sufficient to induce structural changes.

Preliminary Preparation

Owing to the inherent softness of lead cable sheath materials, great care is essential to avoid deformation of the sample to be examined, otherwise there is a danger of obscuring the presence of any porosity and of inducing recrystallisation. For these reasons, manual methods utilising a hack-saw and progressively finer emery papers copiously lubricated with paraffin are preferred to the use of a lathe or microtome.

With this procedure, it is necessary to wash the specimen between changes of emery grade with acetone or another suitable washing medium to prevent emery powder or swarf being carried over to the next stage. Although finer grades of emery are available, these operations are normally terminated at the 3/0 grade, after which the scratches are removed by hand polishing on a jeweller's cloth (Selvyt) loaded with metal polish (Bluebell) for a period of from 1 to 2 minutes. A final abrasive polish is performed mechanically using a pad of jeweller's cloth carrying a diamond powder paste, made up as described in Appendix I. The pad is fitted to an ebonite disc of 5 in. diameter which rotates at about 150 r.p.m. while the specimen traverses across the disc diametrically at a frequency of about 50 c.p.m. Using the lightest pressures practicable, and additions of filtered paraffin to the pad for lubrication, a surface free from scratches visible to the naked eye is obtained in about three minutes.

Etching Techniques

Although a number of etching solutions for lead materials are to be found in published literature (see for example the list given in the Metals Reference Book[7]), the four detailed below have proved suitable for most of the cable sheathing materials studied. These include the alloys in the current issue of British Standards Specification No. 801 and the relatively complex alloys developed in America, containing arsenic, tin and bismuth, with or without tellurium.

TABLE OF ETCHANTS

Solution 1	25% Nitric Acid in Water.
Solution 2	25% Hydrogen Peroxide (100 vol.) and 75% Glacial Acetic Acid. (Worner's Solution,[4] referred to as Chemical Polish.)
Solution 3	Ammonium Molybdate, 16 g., Ammonia, 20 ml., Nitric Acid, 75 ml., and Water, 100 ml.
Solution 4	Ammonium Molybdate, 10 g., Citric Acid, 25 g., and Water 100 ml. (Voce's Solution.)

For a micro-examination, general grain structure characteristics and gross defects may be delineated by swabbing with solution 3 or, less satisfactorily, with solution 1. In the case of cable sheaths, both these solutions may frequently be applied without any preliminary preparation apart from simple degreasing. However, the pitting developed with this technique is

* Communication from the Metallurgical Laboratory, Research Organisation, British Insulated Callender's Cables, Ltd., London, W.12.

† North Hill Plastics Ltd., Manley Court, London, N.16.

‡ Aero Research Ltd., Cambridge.

Fig. 1. 0·85% Sb; 0·05% Cu (Alloy B) × 10
A small chill cast ingot etched with 5% acetic acid in alcohol for 20 minutes, followed by solution 4, showing as-cast structure.

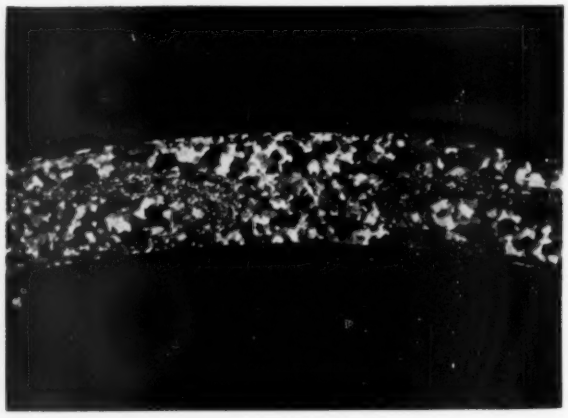

Fig. 2. 0·15% As; 0·1% Sn; 0·1% Bi (Alloy F3) × 6
Ram press extruded pipe etched in solutions 3 and 4, showing charge tongues outlined by regions of small grains.

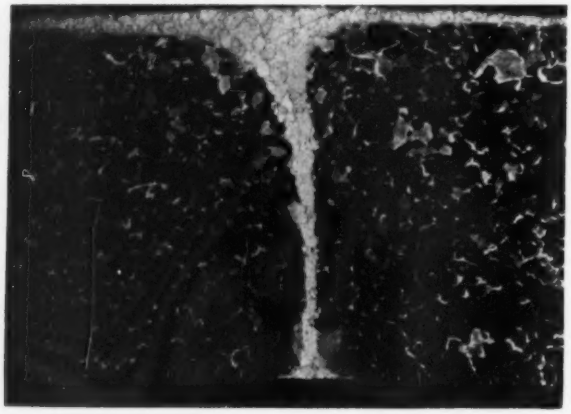

Fig. 3. 0·85% Sb; 0·05% Cu (Alloy B) × 22
Extruded pipe with contamination at weld position which arose due to inadequate cleaning of the press after a previous charge of Alloy E.

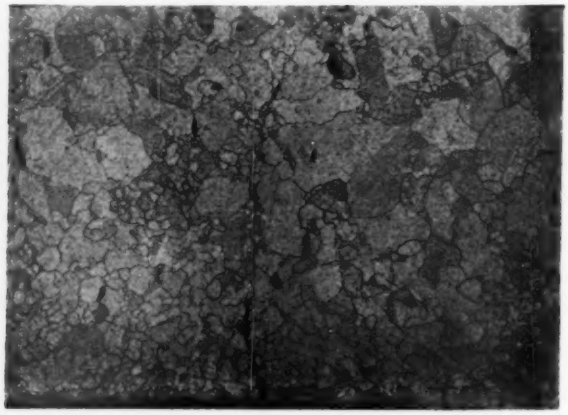

Fig. 4. 0·05% As; 0·1% Sn; 0·1% Bi × 22
Radial cracks developed in coarse grain zone at weld position in an experimental alloy pipe subjected to an internal pressure test.

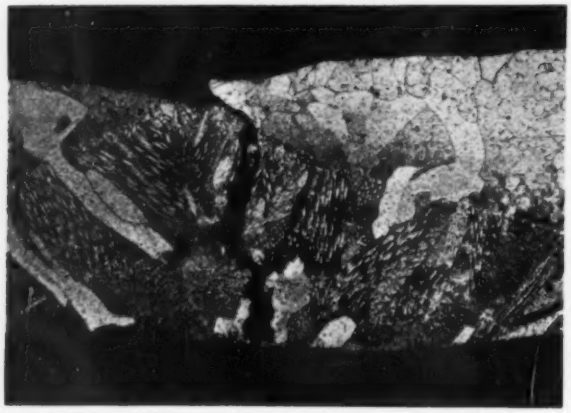

Fig. 5. 0·2% Sb; 0·15% As × 15
Experimental material showing hot-shortness cracking in the as-cast structure of the weld deposit.

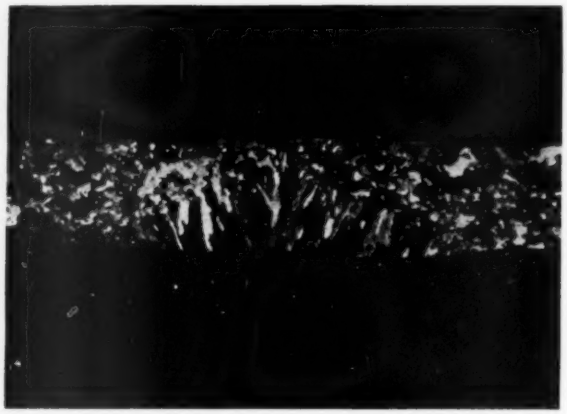

Fig. 6. 0·15% As; 0·1% Sn; 0·1% Bi (Alloy F3) × 7
As Fig. 5 etched in solutions 3 and 4, showing columnar grained weld deposit and incomplete root fusion.

Fig. 7. 0·2% Sb; 0·4% Sn (Alloy E) × 90
Exfoliation at pipe surface associated with segregation of antimony. This defect occurs in ram press extruded pipe and sheath of antimony-bearing alloys and would appear to be due to hot-shortness.

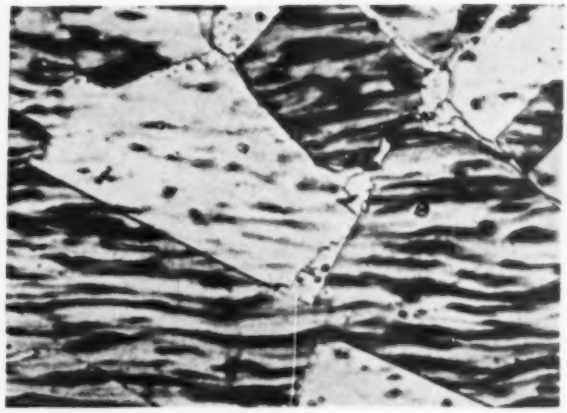

Fig. 8. 0·85% Sb; 0·5% Cu (Alloy B) × 500
As-extruded pipe showing antimony rich flow lines (black) and recrystallised grain boundary regions.

generally too severe to permit detailed examination at magnifications above about × 50.

When a micro-examination is required (magnifications above × 50), the preliminary polishing operations described above must be carried out, followed by the application of solution 2 to remove the last traces of any flowed metal. This solution, however, should be used with care, owing to its vigorous action and marked tendency to produce a pitted surface. Thus, the duration of immersion should not exceed more than a few seconds and, in the case of antimony-bearing alloys, should be restricted to a rapid dip, to avoid the formation of a black surface film.

Solution 2 is useful for outlining the grain boundaries and showing the presence of a second phase, though it is necessary to regard any spherical porosity revealed by this treatment with reserve. The bright surface so obtained can be examined under the microscope without further treatment, but generally the specimen is subsequently immersed in solution 4 for up to 15 seconds. This has the effect of producing a duller surface which is more suitable for photography and at the same time showing up the structure more clearly by differentially etching the grains. The aforegoing is the preferred technique, in that it is particularly useful for revealing fine structural details, such as the mode of precipitation of antimony-rich phases which are darkened by the action of the etch.

Solution 3 is sometimes used as an alternative to the chemical polish (solution 2), with the advantage of better delineation of the structure, and suitability for reproduction work. Unfortunately, its action on arsenic-bearing alloys, and to a lesser extent on antimony-bearing alloys, is rapid and difficult to control. This results in excessive film formation if the time of treatment exceeds a few seconds. In all cases, results from

Fig. 9. 0·85% Sb; 0·05% Cu (Alloy B) × 1750
Section from ram press extruded pipe after creep deformation showing Widmanstätten type precipitation and a light etching grain boundary phase, believed to be micro-segregated antimony and either recrystallised regions or a denuded solid solution, respectively.

Fig. 10. 0·85% Sb; 0·05% Cu (Alloy B) × 250
Dark etching antimony-rich regions and grain boundary precipitate after solution-treatment at 200° C., followed by air cooling.

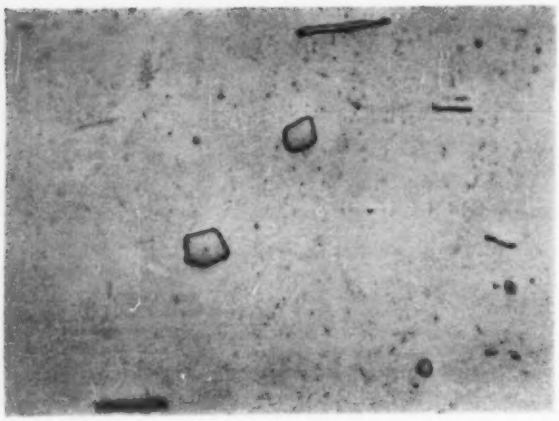

Fig. 11. 0·85% Sb; 0·05% Cu (Alloy B) × 1000
An unetched section showing copper-bearing intermetallic particles.

solution 3 are improved by etching with solution 4, as this effects a brightening of the surface with enhanced contrast.

Results

Some typical photomicrographs of cable sheath materials, prepared as described above, are shown in Figs. 1–13. These photographs were all taken on a standard Vickers Projection Microscope, using either Point-o-lite or carbon arc illumination in conjunction with a yellow or green filter and Kodak 0·250 Rapid Orthochromatic Metallographic plates.

For convenience, the captions in the figures include the nominal alloy content of the material and brief details of the salient features depicted. Unless otherwise stated, the etchants employed were solution 2 followed by solution 4.

Appendix I

Preparation of Diamond Powder Loaded Paste

Composition

Stearic Acid	12·5 g.
Triethanolamine	6·0 ml.
Distilled Water	25·0 ml.
*Diamond Powder (0·1μ particle size) ..	0·5 g.

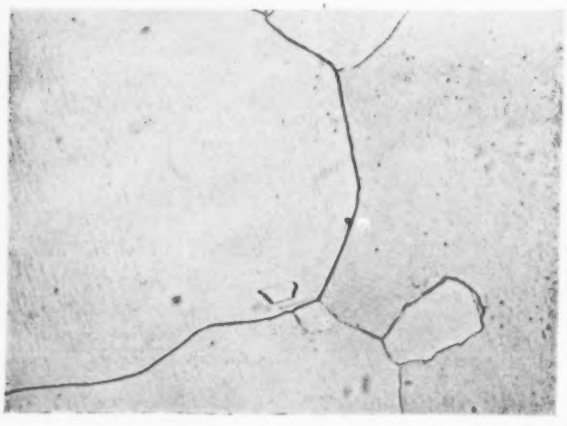

Fig. 13. × 250
Mixture of coarse and isolated small grains in pure lead indicating early stages of recrystallisation.

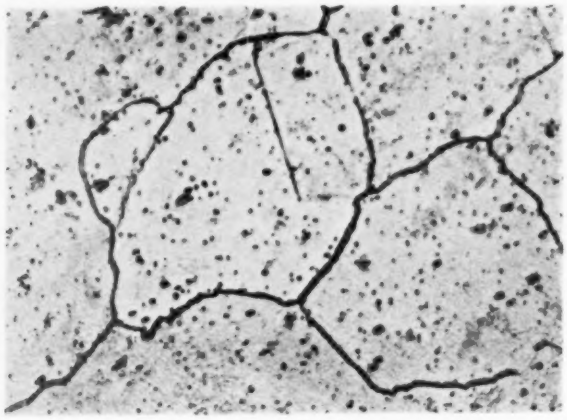

Fig. 12. 0·15% As; 0·1% Sn; 0·1% Bi (Alloy F3) × 750
Transverse section from as-extruded pipe etched with solutions 2 and 3, revealing heavy grain boundary precipitation and dispersed intermetallic particles assumed to be of Sn_3As_2.

Method

The triethanolamine, water and diamond powder are first mixed together and thoroughly stirred to obtain a uniform suspension, which may be facilitated, if desired, by the addition of a small amount of a wetting agent. This mixture is then heated to about 80–90° C. and completed with the stearic acid component at about the same temperature, stirring being continued until a thick paste forms on cooling.

A convenient method of applying the paste to the polishing pad is by means of a hypodermic syringe.

* Supplied by L. M. Van Moppes and Sons, Ltd.

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West's Newcastle Office

Shortly after establishing a Scottish office, West Instrument, Ltd., have now extended their sales and service coverage in the U.K. by opening a Newcastle office, with Mr. W. I. McNall as Manager. The address is: Mostyn, 18 Beechwood Avenue, Gateshead 9, Co. Durham.

Change of Address

Mr. H. Benton, Kelvin Hughes' representative for the North Wales, Cheshire and Lancashire area, has now moved to: 27 Laneside Drive, Bramhall, Cheshire. (Tel.: Bramhall 2841.)

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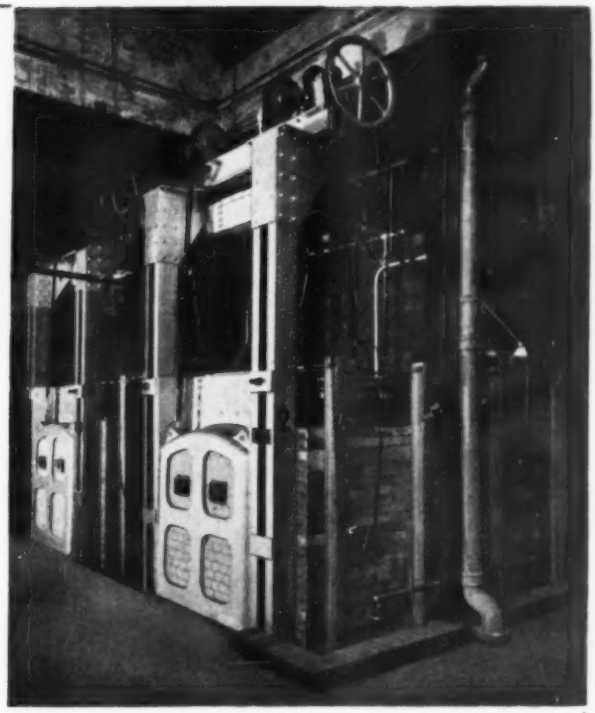

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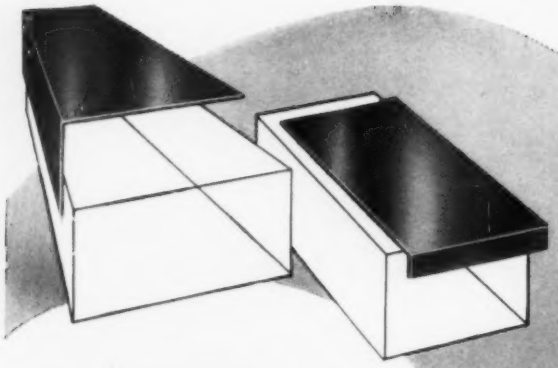

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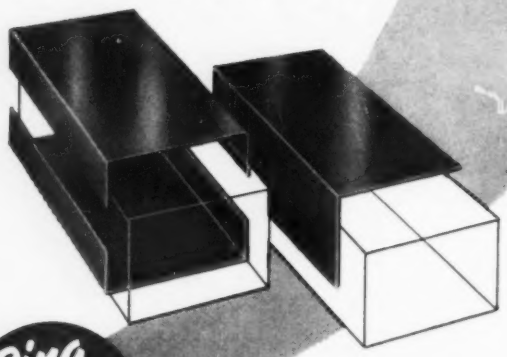

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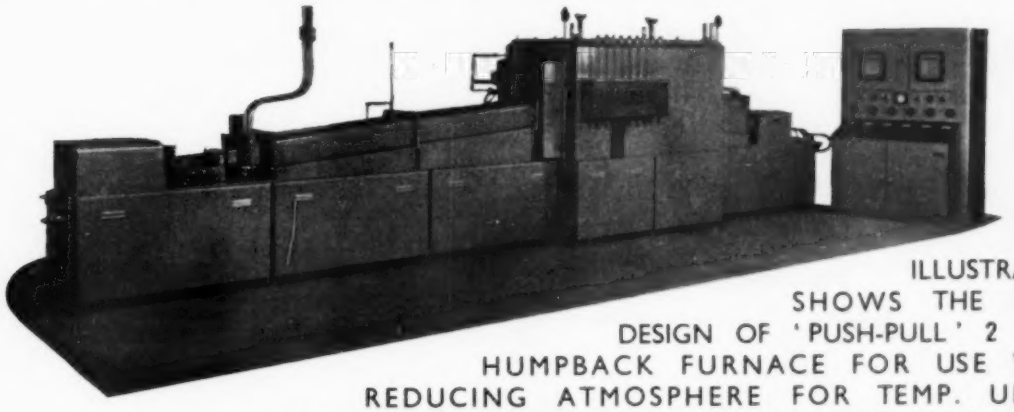

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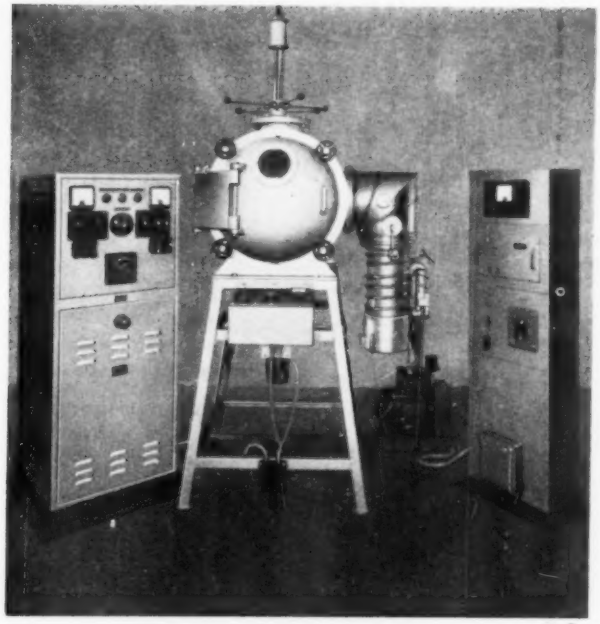

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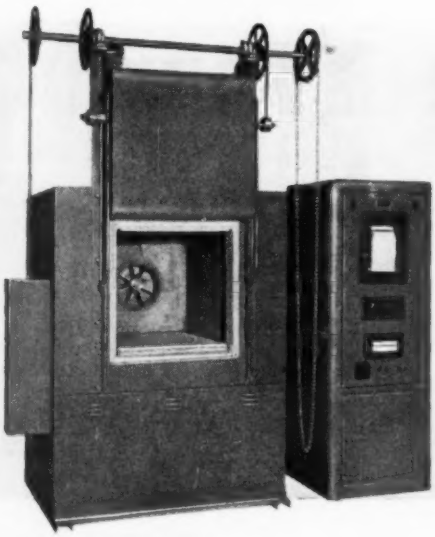

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All applicants should possess an honours degree, or equivalent, in Metallurgy, together with some years' industrial experience. A wide experience of metallographic work is required for post (a) and is desirable, but not essential for post (b).

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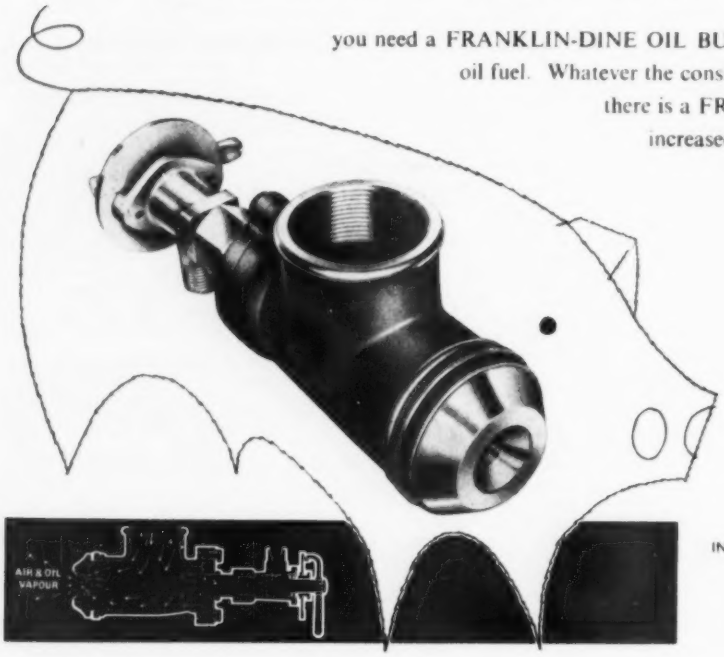

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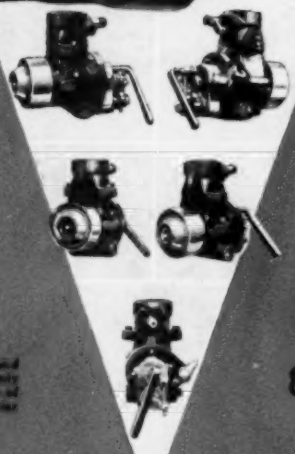

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